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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 = 0.036

wR factor = 0.103

Data-to-parameter ratio = 11.5

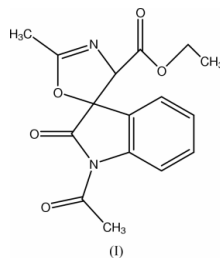
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Ethyl 1-acetyl-2'-methyl-2-oxo-4',5'-dihydro-1*H*-indoline-3-spiro-5'-oxazole-4'-carboxylate

In the title compound, $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_5$, the indoline and oxazole moieties are almost planar, while the five-membered ring in the indoline moiety is slightly distorted towards an envelope conformation. The only intramolecular $\text{C}-\text{H}\cdots\text{O}$ contact forms a six-membered ring. The crystal packing is stabilized by dipole-dipole and van der Waals forces.

Comment

In our recent investigation on photoinduced reactions of 1,2-dicarbonyl compounds with oxazoles (Usman *et al.*, 2002, 2003; Zhang *et al.*, 2003), we have carried out the reaction of 1-acetylisatin with 2-methyl-5-ethoxyoxazole and obtained the title compound, (I), as one of the products. An X-ray crystallographic analysis was undertaken to find the structure and stereochemistry of (I). This compound was derived from an unstable spirooxetane which was formed by photoinduced cycloaddition of the two reactants.



The bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). Those values within the indoline moiety (N1/C1–C8) and oxazole ring (C2/O3/C11/N2/C13) are in good agreement with the corresponding values in related structures (Usman *et al.*, 2002, 2003), except for an elongation of the C8–C13 ($\text{Csp}^3-\text{Csp}^3$) bond of the oxazole ring, due to the bulky substituent attached at C13 (Table 1).

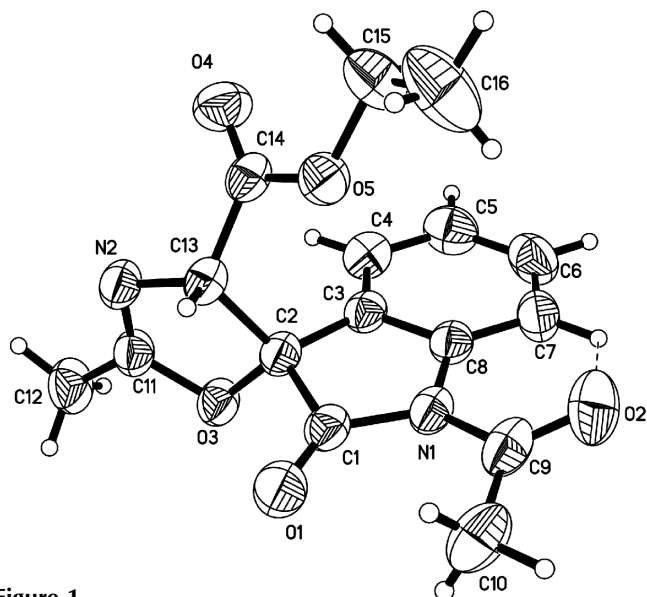
In the title structure (Fig. 1), the indoline moiety is almost planar, with the heterocyclic ring distorted towards an envelope conformation. Atom C2 deviates by 0.134 (3) Å from the O3/C11/N2/C13 plane. The dihedral angle between the five-membered ring and the fused benzene ring is 4.56 (9)°, comparable to the corresponding dihedral angles of 4.2 (1) and 4.6 (2)° (Usman *et al.*, 2003). The acetyl group attached at N1 is twisted around N1–C9 by 18.87 (9)° from the mean plane of the indoline moiety, but is almost perpendicular to the oxazole plane, with a dihedral angle of 84.57 (7)°.

The oxazole ring is slightly non-planar, the largest deviation being 0.223 (9) Å for C2. The oxazole ring and the indoline system are nearly orthogonal, with a dihedral angle of 84.72 (8)° between their planes. The ester substituent attached

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Figure 1

The structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. The intramolecular C—H···O interaction is indicated by a dashed line.

at C13 is almost planar, with C16 deviating 0.4018 Å from the O4/C14/O5/C15 moiety. The ester group makes dihedral angles of 50.28 (10) and 53.16 (8)° with respect to the oxazole ring and the indoline moiety, respectively.

The C14—C13—C2 angle is 112.8 (2)°, implying that the ester group is pushed away from the indoline moiety. The relative orientation of this ester group and indoline system is described by the C14—C13—C2—C3 torsion angle of −14.8 (2)°.

In the crystal structure of the title compound (Fig. 1), there is one intramolecular interaction, C7—H7···O2, forming a six-membered O2—C9—N1—C8—C7—H7 ring. This interaction reinforces the π -conjugation of the acetyl group (Table 2). The crystal packing is stabilized by dipole–dipole and van der Waals forces.

Experimental

The title compound was prepared by the reaction of 1-acetylisatin with an excess of 2-methyl-5-ethoxyoxazole in benzene. The photolysate was separated by silica-gel column chromatography with petroleum ether (b.p. 333–363 K)–ethyl acetate as eluants, giving the title compound as one of the products. A single crystal suitable for X-ray crystallographic analysis was prepared by slow evaporation of solvents from a solution in petroleum ether (b.p. 333–363 K)–acetone (5:1, v/v).

Crystal data

C₁₆H₁₆N₂O₅
 $M_r = 316.31$
 Triclinic, $P\bar{1}$
 $a = 9.348$ (2) Å
 $b = 9.614$ (2) Å
 $c = 9.872$ (2) Å
 $\alpha = 77.015$ (4)°
 $\beta = 63.933$ (3)°
 $\gamma = 88.200$ (4)°
 $V = 774.4$ (3) Å³

$Z = 2$
 $D_x = 1.357$ Mg m^{−3}
 Mo $K\alpha$ radiation
 Cell parameters from 3060 reflections
 $\theta = 2.4$ – 28.3 °
 $\mu = 0.10$ mm^{−1}
 $T = 293$ (2) K
 Block, colourless
 0.80 × 0.30 × 0.20 mm

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.923$, $T_{\max} = 0.980$
 3825 measured reflections

2665 independent reflections
 2290 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\text{max}} = 25.0$ °
 $h = -11 \rightarrow 11$
 $k = -10 \rightarrow 11$
 $l = -11 \rightarrow 11$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.103$
 $S = 1.04$
 2665 reflections
 231 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.1374P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.15$ e Å^{−3}
 $\Delta\rho_{\text{min}} = -0.16$ e Å^{−3}

Table 1

Selected geometric parameters (Å, °).

O3—C11	1.3800 (17)	C1—C2	1.532 (2)
O3—C2	1.4411 (16)	C2—C3	1.492 (2)
N2—C11	1.2617 (19)	C2—C13	1.5758 (19)
N2—C13	1.4543 (18)		
O3—C2—C3	112.75 (11)	C1—C2—C13	110.18 (11)
C3—C2—C1	103.06 (11)	C14—C13—C2	112.86 (11)
C3—C2—C13	118.28 (11)		

Table 2

C—H···O contact (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C7—H7···O2	0.93 (2)	2.37 (2)	2.864 (2)	113 (2)

All H atoms were located in difference Fourier maps and were refined isotropically, except for those attached to C15 and the methyl C atoms, which were positioned geometrically and treated as riding, with C—H distances = 0.93–0.97 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ [$1.2U_{\text{eq}}(\text{C})$ for the H atoms of C15]. Owing to the large fraction of weak data at higher angles, the 2θ maximum was limited to 50°.

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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Ethyl 1-acetyl-2'-methyl-2-oxo-4',5'-dihydro-1H-indoline-3-spiro-5'-oxazole-4'-carboxylate. Erratum

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In the paper by Fun, Wang, Xu, Li & Zhang [*Acta Cryst.* (2003), **E59**, o1827–o1829], two errors are corrected. First, in the second paragraph of the *Comment*, 'C8–C13' on line 6 should read 'C2–C13'. Secondly, in the third paragraph of the *Comment*, 'the O3/C11/N2/C13 plane' on line 4 should read 'the C1/C3/C8/N1 plane'.