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Ethyl 7-oxo-7*H*-indeno[2′,1′:3,4]pyrrolo[2,1-*a*]quinoline-12-carboxylate

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In the title compound, $C_{22}H_{15}NO_3$, the quinoline system is essentially planar, and the mean plane of the indenone ring system is almost coplanar with the quinoline group. The crystal structure is stabilized by inter- and intramolecular C—H···O hydrogen-bonding interactions.

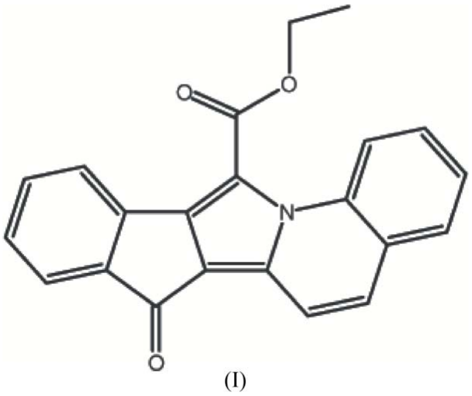
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.128
Data-to-parameter ratio = 12.1

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

Comment

Indolizines annulated at the 1,2-positions are found in several naturally occurring alkaloids with important biological activities, and they are important synthetic targets (Saeva & Luss, 1988). We have recently researched a general and versatile synthesis of 1,2-annulated indolizines by one-pot tandem reactions and obtained the title compound, (I), as one of the products. In order to elucidate the conformation of the compound, we have undertaken an X-ray crystallographic analysis of (I).



(I)

In compound (I) (Fig. 1), the bond lengths and angles are in good agreement with the expected values (Allen *et al.*, 1987). The carboxylate group is twisted, with a dihedral angle between the five-membered N-heterocyclic ring and the carboxylate group of 39.22 (13)°.

In the packing of compound (I), the molecules are linked into layers (Fig. 2) by C—H···O intermolecular hydrogen-bonding interactions. Details of these and of the intramolecular hydrogen bonds are given in Table 2.

Experimental

A mixture of *N*-(carboxyethyl)quinolinium salts (1.1 mmol), 2,3-dichloroindenone (1 mmol) and potassium carbonate (3.5 mmol) in MeCN (15 ml) was heated at 323 K for 24 h with magnetic stirring. Compound (I) was isolated by column chromatography of the reaction mixture on silica gel after evaporation of the solvent, in 28% yield. Single crystals of (I) were obtained by slow evaporation of a solution in petroleum ether–ethyl acetate (3:1).

Crystal data

$C_{22}H_{15}NO_3$
$M_r = 341.35$
Triclinic, $P\overline{1}$
$a = 7.5210$ (15) Å
$b = 9.4560$ (19) Å
$c = 12.411$ (3) Å
$\alpha = 99.33$ (3)°
$\beta = 98.15$ (3)°
$\gamma = 108.13$ (3)°
$V = 810.2$ (4) Å^3
$Z = 2$
$D_x = 1.399$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*XCAD4*; Harms & Wocadlo, 1995)
$T_{\min} = 0.872$, $T_{\max} = 0.973$
3086 measured reflections
2847 independent reflections
1941 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\max} = 25.0$°
3 standard reflections every 200 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.129$
$S = 1.00$
2847 reflections
236 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.057P)^2 + 0.2168P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.025 (3)

Table 1
Selected torsion angle (°).

C21—O1—C20—O2	5.8 (4)	C9—N—C5—C4	13.4 (3)
C3—C2—C1—C6	−2.3 (4)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*A*···O1	0.93	2.42	2.909 (3)	113
C6—H6*A*···O3$^{\rm i}$	0.93	2.58	3.243 (3)	128
C16—H16*A*···O2	0.93	2.52	3.220 (3)	132
C21—H21*A*···O2$^{\rm ii}$	0.97	2.54	3.313 (3)	136

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

The H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with $U_{\rm iso}(H) = 1.2U_{\rm eq}(C)$, or $U_{\rm iso}(H) = 1.5U_{\rm eq}(C_{\rm methyl})$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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* and *PLATON* (Spek, 2003).

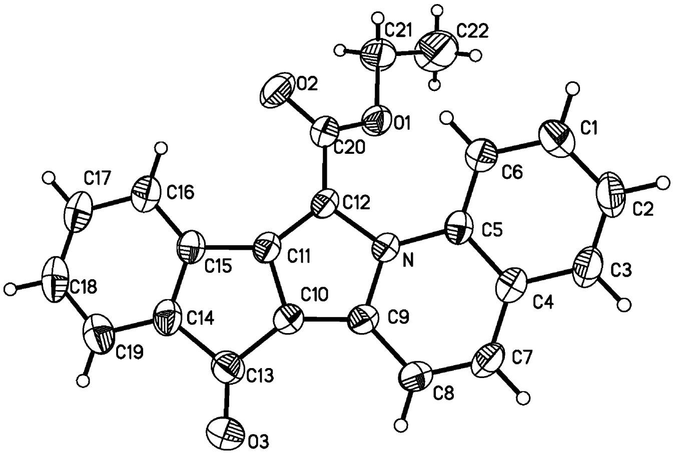


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

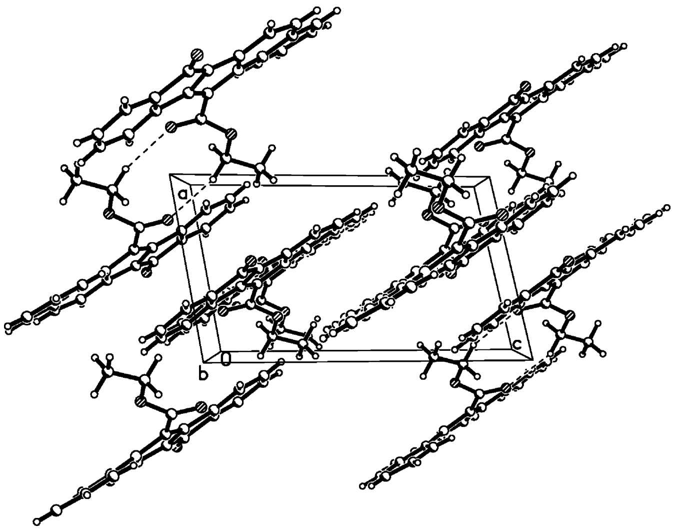


Figure 2
A packing diagram of (I). Dashed lines indicate intermolecular hydrogen bonds.

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Saeva, F. D. & Luss, H. R. (1988). *J. Org. Chem.* **53**, 1804–1806.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

o760 Liu *et al.* • $C_{22}H_{15}NO_3$ *Acta Cryst.* (2007). E**63**, o759–o760