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Key indicators

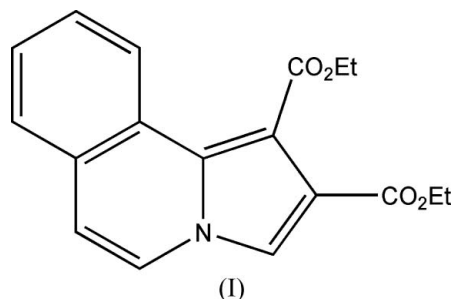
Single-crystal X-ray study
 $T = 297$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.055
 wR factor = 0.149
Data-to-parameter ratio = 13.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Diethyl pyrrolo[2,1-*a*]isoquinoline-1,2-dicarboxylate

In the title compound, $\text{C}_{18}\text{H}_{17}\text{NO}_4$, the indolizine unit is not completely planar. Intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions help to stabilize the crystal structure.

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Comment

Indolizines are electron-rich heterocycles and 3-unsubstituted indolizine is especially important in the family of indolizines, as the highest electronic population in the π -excessive heterocycle occurs on C3, which allows many electrophilic substitutions (Reid *et al.*, 1979). The structure of the title compound, (I), has been determined as we use 3-unsubstituted indolizines as electrophilic-substituted reagents.



The bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987), and comparable with those in related structures (Usman *et al.*, 2002). In the title structure (Fig. 1), the indolizine unit is not completely planar, the dihedral angle between the rings being 1.19° . The dihedral angle between the two carboxylate groups is 77.62° . One of the carboxylate groups (C2/C14/O3/O4) is almost coplanar with the indolizine unit (see torsion angles in Table 1).

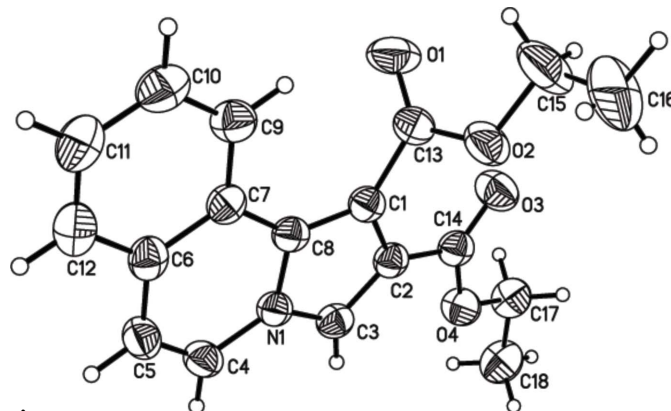


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

The crystal structure of (I) exhibits two intermolecular hydrogen-bond interactions (Table 2), which help to stabilize the crystal structure.

Experimental

A suspension of 2-(carboxymethyl)isoquinolinium bromide (10 mmol), diethyl maleate (50 mmol) and Et₃N (1.5 ml) in toluene (80 ml) was stirred at 363 K for 2 h (monitored by thin-layer chromatography). It was then filtered, and the organic layer was evaporated and chromatographed to give (I) (yield 78%) (Zhang *et al.*, 2000). Single crystals suitable for X-ray crystallographic analysis were obtained by recrystallization from acetone.

Crystal data

C ₁₈ H ₁₇ NO ₄	$V = 802.2 (3) \text{ \AA}^3$
$M_r = 311.33$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.289 \text{ Mg m}^{-3}$
$a = 7.5960 (15) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.920 (2) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 11.451 (2) \text{ \AA}$	$T = 297 (2) \text{ K}$
$\alpha = 92.27 (3)^\circ$	Block, colorless
$\beta = 98.09 (3)^\circ$	$0.40 \times 0.30 \times 0.30 \text{ mm}$
$\gamma = 109.42 (3)^\circ$	

Data collection

Enraf–Nonius CAD-4 diffractometer	2812 independent reflections
$\omega/2\theta$ scans	2060 reflections with $I > 2\sigma(I)$
Absorption correction: ψ scan (XCAD4; Harms & Wocadlo, 1995)	$R_{\text{int}} = 0.019$
$T_{\text{min}} = 0.964$, $T_{\text{max}} = 0.973$	$\theta_{\text{max}} = 25.0^\circ$
3046 measured reflections	3 standard reflections every 200 reflections
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0708P)^2 + 0.3401P]$
$R[F^2 > 2\sigma(F^2)] = 0.055$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.149$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$
2812 reflections	$\Delta\rho_{\text{min}} = -0.39 \text{ e \AA}^{-3}$
203 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.058 (7)

Table 1

Selected torsion angles ($^\circ$).

C8–C1–C13–O2	104.9 (3)	C7–C8–N1–C3	178.66 (18)
C1–C2–C14–O4	179.27 (19)		

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C5–H5A \cdots O3 ⁱ	0.93	2.59	3.451 (3)	154
C10–H10A \cdots O1 ⁱⁱ	0.93	2.53	3.415 (3)	157

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

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

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).

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. Enraf–Nonius, Delft, The Netherlands.
- Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
- Reid, D. H., Webster, T. G. & McKenzie, S. (1979). *J. Chem. Soc. Perkin Trans. 1*, pp. 2334–2339.
- Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Usman, A., Li, Y., Zhang, Y., Fun, H.-K. & Xu, J.-H. (2002). *Acta Cryst.* **E58**, o1427–o1429.
- Zhang, L. D., Liang, F., Sun, L. Z., Hu, Y. F. & Hu, H. W. (2000). *Synthesis*, pp. 1733–1737.