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Dimethyl 3-benzoyl-7-(*N*,*N*-dimethylamino)-indolizine-1,2-dicarboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.134
Data-to-parameter ratio = 16.6

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

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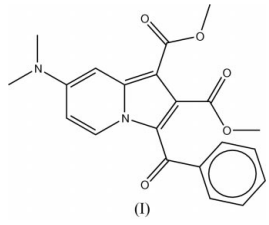
In the title compound, $C_{21}H_{20}N_2O_5$, the molecular packing is influenced by weak intermolecular C—H···O and C—H···π interactions. The carboxylate groups are oriented perpendicular to each other and one of them is almost coplanar with the indolizine moiety.

Comment

Heterocyclic compounds, such as indolizines, are important bioactive compounds that have a wide range of applications in biology, pharmacology and agrochemistry (Wu & Chen, 2003, and references therein). The synthesis of biologically active indolizines (Gubin *et al.*, 1992) continues to attract the attention of organic chemists (Bora *et al.*, 2003, and references therein), because they are important as potential central nervous system depressants, calcium entry blockers, cardiovascular agents, spectral sensitizers and novel dyes (Katritzky *et al.*, 1999, and references therein). In view of these important attributes of indolizine derivatives, we report here the crystal structure of the title compound, (I).



(I)

A perspective view of the molecule of (I), with the atomic numbering scheme, is shown in Fig. 1. The bond lengths and angles in (I) are comparable with those in related structures (Pritchard, 1988; Usman *et al.*, 2002; Hema *et al.*, 2003). The dihedral angle between the planes of the indolizine moiety and the benzoyl ring is 58.13 (5)°. The planes of the 1- and 2-carboxylate groups are oriented at angles of 5.97 (9) and 72.05 (7)°, respectively, with respect to the plane of the indolizine moiety. The carboxylate groups are approximately perpendicular to each other.

In the crystalline state, atom C5 is involved in a weak intermolecular C—H···O interaction (Table 1) with atom O16 of a centrosymmetrically related molecule, thus forming an $R_2^2(12)$ motif (Bernstein *et al.*, 1995). Atom C20 acts as a donor in a weak intermolecular C—H···O interaction with carbonyl atom O10 of an adjacent molecule. This interaction links the molecules into a chain that runs parallel to the *c* axis and has a graph-set motif of *C*(11). In addition, the phenyl ring (*Cg*1) and the six-membered ring of the indolizine moiety are

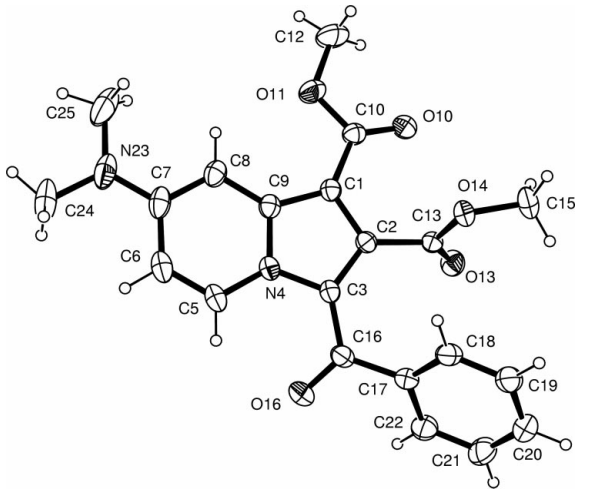


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by spheres of arbitrary radii.

involved in weak intermolecular C—H···π interactions (Table 1).

Experimental

A mixture of 4-dimethylaminopyridinium-1-phenacylide (1.4 mmol), dimethyl acetylenedicarboxylate (1.6 mmol) and potassium carbonate (1.6 mmol) in dimethylformamide (30 ml) was kept at room temperature overnight. The insoluble materials were removed by filtration, and the filtrate was extracted with an ethyl acetate–dilute HCl mixture. The organic layer was evaporated and chromatographed to give (I), which was recrystallized from ethyl acetate (yield 0.29 g, 55%; m.p. 474–476 K).

Crystal data

$C_{21}H_{20}N_2O_5$
M_r = 380.39
Monoclinic, $P2_1/c$
a = 8.6333 (9) Å
b = 27.515 (3) Å
c = 7.9705 (9) Å
β = 99.781 (2)°
V = 1865.8 (3) Å^3
Z = 4
D_x = 1.354 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4242 reflections
θ = 2.4–27.5°
μ = 0.10 mm^{-1}
T = 293 (2) K
Prism, yellow
0.30 × 0.25 × 0.18 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
11 309 measured reflections
4265 independent reflections
3301 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 28.0°
$h = -11 \rightarrow 11$
$k = -35 \rightarrow 33$
$l = -8 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.134
S = 1.03
4265 reflections
257 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0689P)^2 + 0.3296P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Geometry of C—H···O and C—H···π interactions (Å, °).

*Cg*1 is the centroid of the phenyl ring and *Cg*2 is the centroid of the six-membered ring of the indolizine moiety.

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O16^{i}	0.93	2.37	3.142 (2)	140
C20—H20···O10^{ii}	0.93	2.59	3.501 (2)	165
C12—H123···*Cg*1^{iii}	0.96	2.93	3.721 (3)	141
C24—H243···*Cg*2^{iv}	0.96	2.79	3.677 (2)	154

Symmetry codes: (i) $-x, -y, -z$; (ii) $x-1, \frac{1}{2}-y, z-\frac{1}{2}$; (iii) $1+x, y, 1+z$; (iv) $1-x, -y, -z$.

The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å), with U_{iso}(H) = 1.5U_{eq}(C), but were allowed to rotate freely about the C—C bond. All other H atoms were placed in idealized positions (C—H = 0.93 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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