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Dimethyl 7-(*N*,*N*-dimethylamino)-3-(4-methylbenzoyl)indolizine-1,2-dicarboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.057
wR factor = 0.156
Data-to-parameter ratio = 15.2

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

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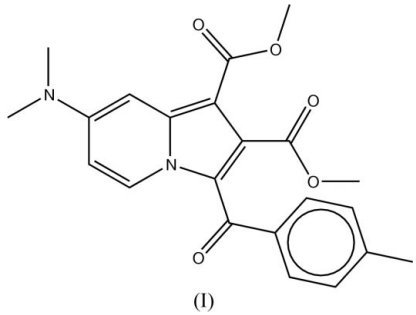
In the title compound, $C_{22}H_{22}N_2O_5$, there are two molecules in the asymmetric unit. In each of them, the carboxylate groups are oriented perpendicular to each other and one of the carboxylate groups is almost coplanar with the indolizine moiety. In the solid state, weak intermolecular C—H···O contacts are observed.

Comment

Chemists are attracted by indolizines and their derivatives because of their importance as pharmaceutical drugs, such as potential central nervous system depressants, cardiovascular agents, calcium entry blockers, spectral sensitizers and novel dyes (Hema *et al.*, 2003, and references therein). Indolizine derivatives such as 1-carboxymethyl-3-(4-chlorobenzoyl)-7-methoxy-2-methylindolizine, 3-acetyl(benzoyl)-1-carboxyethyl)indolizine and 3-carboxymethyl-1-(4-chlorobenzoyl)-7-methoxy-2-methylindolizine exhibit *anti*-inflammatory activity (Casagrande *et al.*, 1971). 3–Benzoylindolizine-1-acetic acid exhibits an auxin-like activity (Carbellini *et al.*, 1968). In view of the these important attributes, the structure determination of the title compound, (I), was performed.



(I)

The asymmetric unit of (I) contains two crystallographically independent molecules (*A* and *B*). A view of the two independent molecules, including the atomic numbering scheme, is shown in Fig. 1. The overall weighted r.m.s. fit of molecules *A* and *B* is 0.095 Å. The corresponding bond lengths and angles in the independent molecules agree with each other and are comparable to those in related structures (Hema *et al.*, 2003, 2004). The planes of the 1- and 2-carboxylate groups are oriented at angles of 4.52 (6) [3.15 (10)° for molecule *B*] and 72.05 (7)° [74.67 (14)° for molecule *B*], respectively, with respect to the plane of the indolizine moiety. The corresponding dihedral angles in a related structure (Hema *et al.*, 2004) are 5.97 (9) and 72.05 (7)°, respectively. The carboxylate groups are oriented perpendicular to each other. The dihedral angle between the planes of the indolizine moiety and the

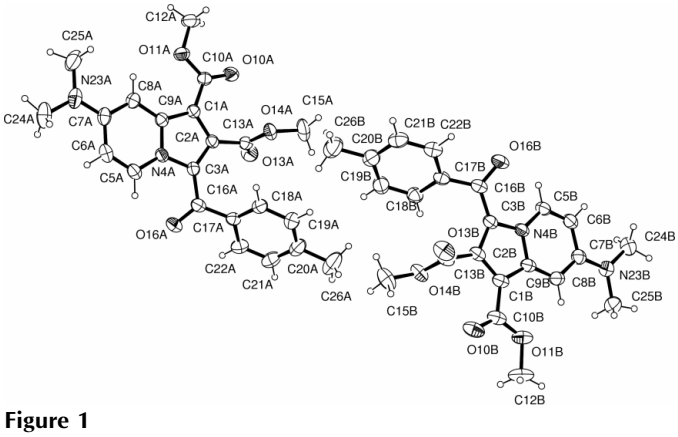


Figure 1
The two independent molecules of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by circles of arbitrary radii.

benzoyl ring is 59.13 (6)° [58.31 (6)° for molecule *B*], while the corresponding angle in a related structure (Hema *et al.*, 2004) is 58.13 (5)°.

In the crystal structure, atom C5*B* of molecule *B* acts as a donor for a weak intermolecular C—H···O interaction with carbonyl atom O16*B* of a symmetry-related molecule at $(1-x, -y, -z)$, leading to an $R_2^2(12)$ motif (Bernstein *et al.*, 1995). Atom C15*B* of molecule *B* is involved in a weak intermolecular C—H···O interaction with atom O10B of a symmetry-related molecule at $(2-x, 1-y, 1-z)$ and has a graph-set motif of $R_2^2(16)$ (Table 1). Atom C25*B* acts as a donor for a weak intermolecular C—H···O interaction with carbonyl atom O16*A* of molecule *A* of an adjacent molecule.

Experimental

A mixture of 4-dimethylaminopyridinium-1-(4-methyl)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 47%, m.p. 443–445 K).

Crystal data

$C_{22}H_{22}N_2O_5$
M_r = 394.42
Triclinic, $P\overline{1}$
a = 8.050 (5) Å
b = 16.942 (11) Å
c = 17.094 (11) Å
α = 118.555 (10)°
β = 95.033 (12)°
γ = 97.082 (11)°
V = 2003 (2) Å^3
Z = 4
D_x = 1.308 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3068 reflections
θ = 2.4–24.5°
μ = 0.09 mm^{-1}
T = 293 (2) K
Prism, yellow
0.28 × 0.20 × 0.15 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
12 347 measured reflections
8081 independent reflections
5203 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 28.1°
$h = -10 \rightarrow 9$
$k = -22 \rightarrow 17$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.156
S = 1.03
8081 reflections
533 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2 + 0.2476P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
C—H···O interactions (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C5*B*—H52···O16*B*^{i}	0.93	2.45	3.154 (3)	132
C15*B*—H155···O10*B*^{ii}	0.96	2.52	3.417 (4)	156
C25*B*—H256···O16*A*^{iii}	0.96	2.60	3.531 (4)	164

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

The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å), with U_{iso}(H) values of $1.5U_{iso}$(C), but were allowed to rotate freely about the C—C bond. All remaining H atoms were placed in idealized positions (C—H = 0.93 Å) and constrained to ride on their parent atoms, with U_{iso}(H) values of $1.2U_{iso}$(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 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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