Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.101
Data-to-parameter ratio = 21.4

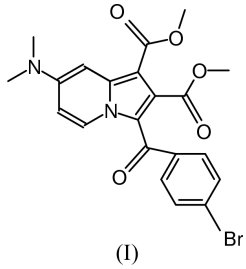
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 10 March 2004
Accepted 24 March 2004
Online 31 March 2004

In the title compound, $C_{21}H_{19}BrN_2O_5$, the planes of the two methoxycarbonyl moieties are oriented at angles of 7.70 (6) and 69.09 (6)° with respect to that of the indolizine ring. In the solid state, the molecules are held together by weak intermolecular C—H···O and C—H···Br interactions which form chain and centrosymmetric ring motifs.

Comment

The synthesis of biologically active indolizine derivatives continues to attract the attention of organic chemists, because they are important as potential central nervous system depressants, calcium entry blockers, cardiovascular agents, spectral sensitizers and novel dyes (Gubin *et al.*, 1992; Bora *et al.*, 2003). Indolizines have also been tested as anti-mycobacterial agents against mycobacterial tuberculosis (Gundersen *et al.*, 2003).



(I)

The structural investigation of the title compound, (I) (Fig. 1), has been undertaken as a part of our study on the conformational changes caused by different substituents at various positions on the indolizine ring system. The bond lengths and angles in (I) are comparable with those in related structures (Pritchard, 1988; Hema *et al.*, 2003). The non-H atoms of (I) common to two related indolizine derivatives, *viz*. 3-(4-chlorobenzoyl)-7-(*N*,*N*-dimethylamino)-1-phenylindolizine and 3-(2,4-dichlorobenzoyl)-7-(*N*,*N*-dimethylamino)-1-

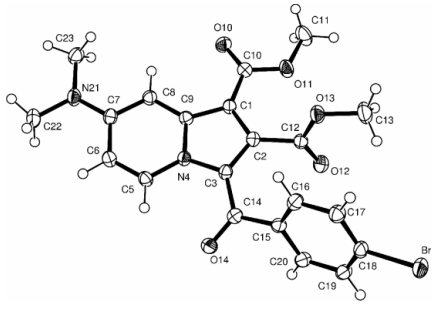


Figure 1
View of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary radii.

phenylindolizine (Hema *et al.*, 2003), were superimposed on the corresponding atoms of these latter compounds and the r.m.s. deviations were found to be 0.958 and 0.965 Å, respectively. The planes of the two methoxycarbonyl moieties deviate from the plane of the indolizine ring to different extents, the angles between the latter plane and those of the C1/C10/O10/O11/C11 and C2/C12/O12/O13/C13 moieties being 7.70 (6) and 69.09 (6)°, respectively. The dihedral angle between the planes of the bromophenyl ring and the indolizine ring system is 68.34 (5)°, while the plane of the carbonyl moiety C2/C14/O14/C15 lies roughly between these two orientations and makes angles of 59.39 (6) and 20.69 (5)°, respectively, with the planes of the bromophenyl ring and the indolizine ring system.

The crystal packing is stabilized by a number of weak intermolecular C—H···O and C—H···Br interactions (Table 1). The C—H···Br interaction links pairs of molecules across centres of inversion to give the ring motif $R_2^2(24)$ (Bernstein *et al.*, 1995).

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C13—H133···O12^{i}	0.98	2.45	3.419 (3)	170
C17—H17···O10^{ii}	0.95	2.50	3.368 (2)	152
C19—H19···O10^{iii}	0.95	2.31	3.249 (2)	172
C23—H233···O14^{iv}	0.98	2.44	3.167 (2)	131
C13—H131···Br^{v}	0.98	2.84	3.480 (2)	123

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

Experimental

A mixture of 4-dimethylaminopyridinium 1-(4-bromo)phenacylide (1.4 mmol), dimethylacetylene dicarboxylate (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. 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

$C_{21}H_{19}BrN_2O_5$
M_r = 459.29
Triclinic, $P\overline{1}$
a = 7.5238 (2) Å
b = 10.2817 (2) Å
c = 13.8084 (2) Å
α = 69.467 (1)°
β = 79.837 (1)°
γ = 77.741 (1)°
V = 971.42 (3) Å^3
Z = 2
D_x = 1.570 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 29 442 reflections
θ = 2.0–30.0°
μ = 2.15 mm^{-1}
T = 160 (2) K
Prism, yellow
0.30 × 0.23 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.542, T_{max} = 0.641
25900 measured reflections
5684 independent reflections
4679 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 30.0°
$h = -10 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -19 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.101
S = 1.03
5684 reflections
266 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 0.3259P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

The methyl H atoms were constrained to an ideal geometry (C—H = 0.98 Å), with U_{iso} values of $1.5U_{eq}$(C), but were allowed to rotate freely about the C—C bond. All remaining H atoms were placed in idealized positions (C—H = 0.95 Å) and constrained to ride on their parent atoms, with U_{iso}(H) values of $1.2U_{eq}$(C).

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); 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 (Version 1.07; Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

Thanks are due to the Council of Scientific and Industrial Research (CSIR), India, for the award of a Senior Research Fellowship (2001–2004) to KS. RH thanks the UGC, India, for the award of a Minor Research Project.

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