

1,3-Dibenzoyl-5,8-dimethoxy-2-phenyl-5,8-dihydroindolizine

Hoong-Kun Fun,^{a*} Yun Li,^b
Jian-Hua Xu,^b Xue-Mei Li^c and
Shu-Sheng Zhang^c

^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, ^bDepartment of Chemistry, Nanjing University, 210093 Nanjing, People's Republic of China, and ^cCollege of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, 266042 Qingdao, Shandong, People's Republic of China

Correspondence e-mail: hkfun@usm.my

Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$

R factor = 0.044

wR factor = 0.143

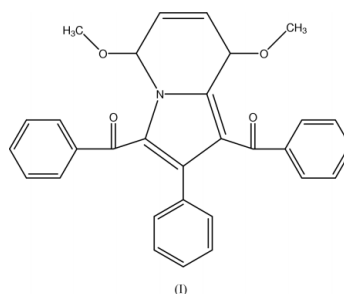
Data-to-parameter ratio = 15.5

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

In the title compound, $\text{C}_{30}\text{H}_{25}\text{NO}_4$, the indolizine moiety is not completely planar, the six-membered ring being distorted towards an envelope conformation. The benzoyl and phenyl substituents are each almost planar. The crystal structure is stabilized by dipole–dipole and van der Waals forces.

Comment

Indolizines are electron-rich heterocycles with very low oxidation potential. We have recently investigated the reactions of indolizines with the strong one-electron oxidant ceric ammonium nitrate (CAN). The title compound, (I), was obtained as one of the products in this study. An X-ray crystallographic analysis was undertaken to find the structure and stereochemistry of (I).



The bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). In the title structure (Fig. 1), the indolizine moiety is not completely planar, the six-membered ring being distorted towards an envelope conformation. The deviation of N1 from the C3–C7 mean plane is 0.218 (8) Å. The two methoxy substituents attached at C3 and C6 are on opposite sides of the indolizine mean plane, due to the sp^3 hybridization state of atoms C3 and C6. The benzoyl substituents attached at C2 and C8 are each almost planar, although O3 and O4 deviate by 0.667 (9) and -0.567 (7) Å respectively, from these two planes. The angle between the mean planes of these two benzoyl moieties is 52.00 (8)°. The benzene ring attached at C1 is nearly perpendicular to the C10–C15 phenyl ring, the dihedral angle being 85.82 (8)°, while the corresponding angle is 42.30 (9)° with respect to the C23–C28 phenyl ring.

The crystal structure is stabilized by dipole–dipole and van der Waals forces.

Experimental

The title compound was prepared by the reaction of 1,3-dibenzoyl-2-phenylindolizine with ceric ammonium nitrate (CAN) in a methanol solution at room temperature, followed by silica-gel chromatographic separation of the reaction mixture with petroleum ether (b.p. 333–363 K)–ethyl acetate as eluants. A single crystal suitable for X-ray crystallographic analysis was prepared by slow evaporation of a petroleum ether (b.p. 333–363 K) acetone solution (3:1, v/v).

Received 2 October 2003

Accepted 14 October 2003

Online 8 November 2003

Crystal data

$C_{30}H_{25}NO_4$
 $M_r = 463.51$
 Orthorhombic, *Pbca*
 $a = 12.8820$ (6) Å
 $b = 12.1160$ (5) Å
 $c = 30.7987$ (13) Å
 $V = 4807.0$ (4) Å³
 $Z = 8$
 $D_x = 1.281$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 7716
 reflections
 $\theta = 2.4$ – 28.3°
 $\mu = 0.09$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 $0.60 \times 0.56 \times 0.54$ mm

Data collection

Siemens SMART CCD area-
 detector diffractometer
 ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.951$, $T_{\max} = 0.956$
 28443 measured reflections

5925 independent reflections
 4883 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$
 $\theta_{\text{max}} = 28.3^\circ$
 $h = -17 \rightarrow 10$
 $k = -16 \rightarrow 15$
 $l = -35 \rightarrow 40$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.143$
 $S = 0.79$
 5925 reflections
 382 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.1093P)^2 + 2.1064P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O1—C6	1.4357 (16)	C3—C4	1.4968 (19)
O2—C3	1.4205 (15)	C5—C6	1.5002 (18)
O2—C3—N1	110.33 (10)	O1—C6—C5	111.72 (10)
O2—C3—C4	112.45 (11)	O1—C6—C7	111.44 (10)

All H atoms were located in difference Fourier maps and were refined isotropically, except for those attached to C19, C20, C26 and the methyl C atoms, which were fixed geometrically and treated as riding, with C—H = 0.93–0.96 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ [$1.5U_{\text{eq}}(\text{C})$ for the methyl H atoms].

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve

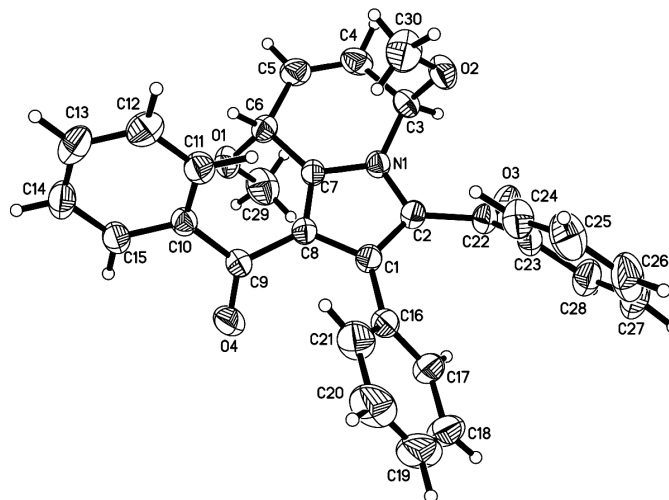


Figure 1

The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

structure: SHELXTL (Bruker, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

The authors thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 305/PFIZIK/610961.

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.
 Bruker (1997). SHELXTL. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
 Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Siemens (1996). SMART and SAINT. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.