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Key indicators

Single-crystal X-ray study
T = 294 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.047
wR factor = 0.139
Data-to-parameter ratio = 13.9

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

Ethyl 8-benzoyl-5-oxo-7-phenyl-1,2,3,5,6,7-hexahydroimidazo[1,2-a]pyridine-6-carboxylate

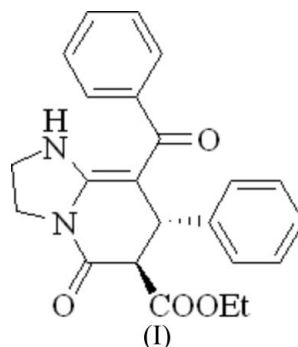
The geometric parameters for the title compound, $\text{C}_{23}\text{H}_{22}\text{N}_2\text{O}_4$, are normal. The molecules form dimeric associations via inversion-generated pairs of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Received 25 May 2006

Accepted 1 June 2006

Comment

5-Oxoimidazo[1,2-a]pyridine derivatives are potentially biologically active compounds. Heterocyclic ketene amins (HKAs) are useful intermediates for the preparation of these compounds. We have reported some examples of HKAs reacting with α,β -unsaturated compounds to yield 5-oxoimidazo[1,2-a]pyridine derivatives (Huang & Liu, 1986). Compound (I) (Fig. 1) (Table 1) resulted from a Michael addition reaction (see *Experimental*). A $^1\text{H}-^1\text{H}$ nuclear Overhauser effect experiment failed to provide unambiguous evidence for the determination of a *Z* or *E* configuration about the $\text{C}1=\text{C}16$ double bond and its crystal structure was determined to resolve this ambiguity.



Compound (I) is chiral (in the molecule of the arbitrarily chosen asymmetric unit, C5 has an *R* configuration and C9 has an *S* configuration), but crystal symmetry generates a racemic mixture of the two enantiomers.

The six-membered $\text{C}1/\text{N}2/\text{C}4/\text{C}5/\text{C}9/\text{C}16$ ring in (I) adopts a twist boat conformation; the torsion angle $\text{N}2-\text{C}4-\text{C}5-\text{C}9$ is $-34.7(3)^\circ$ and $\text{N}2-\text{C}1$ and $\text{C}16-\text{C}9$ is $3.2(2)^\circ$. The $\text{H}5-\text{C}5-\text{C}9-\text{H}9$ dihedral angle of 74° is consistent with the value (64.1°) calculated by application of the Karplus equation on the basis of the NMR coupling constant of 1.9 Hz for these H atoms.

The $\text{N}1-\text{H}1$ group participates in a bifurcated $\text{N}-\text{H}\cdots(\text{O},\text{O})$ interaction: an intramolecular hydrogen bond to O4 (Table 2) and an intermolecular bond to a second, inversion symmetry-generated, O4 species in an adjacent molecule (bond angle sum about $\text{H}1 = 350^\circ$) (Fig. 2). The molecular packing is shown in Fig. 3.

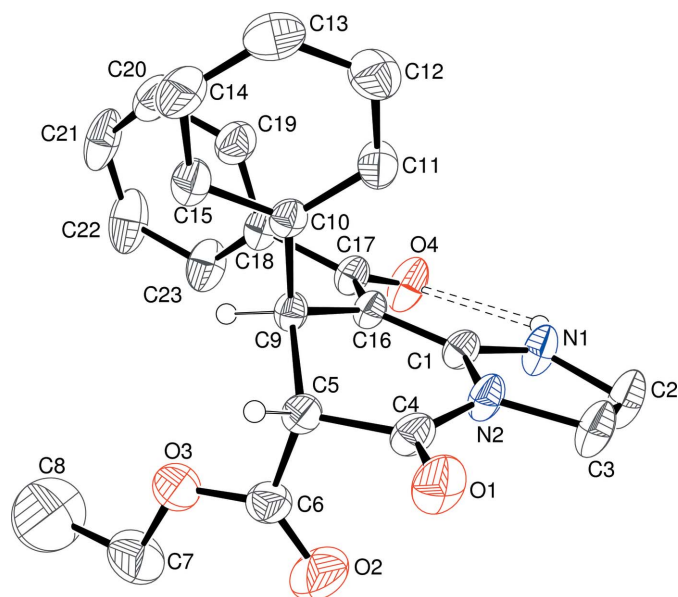


Figure 1
A view of (I), showing 40% displacement ellipsoids (arbitrary spheres for the H atoms). All H atoms except H1, H5 and H9 have been omitted for clarity. The dashed line indicates a hydrogen bond.

Experimental

A solution of 2-(benzoylmethylene)imidazolidine (1 mmol) (Huang & Wang, 1992) and diethyl 2-benzylidene malonate (1 mmol) derived from benzaldehyde (Breukelman *et al.*, 1981) in dioxane (10 ml) was stirred at 363 K for 12 h and then refluxed for 36 h. After removal of the solvent, the desired product was separated by flash chromatography (ethyl acetate–petroleum ether, 1.6:1) to yield the title compound, (I), in 71% yield (m.p. 440 K). Crystals of (I) were recrystallised from ethyl acetate–petroleum ether (ca 1:6).

Crystal data

$C_{23}H_{22}N_2O_4$
 $M_r = 390.43$
 Monoclinic, $P2_1/c$
 $a = 9.644$ (3) Å
 $b = 11.937$ (3) Å
 $c = 17.998$ (5) Å
 $\beta = 90.691$ (5)°
 $V = 2071.8$ (10) Å³

$Z = 4$
 $D_x = 1.252$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 294$ (2) K
 Block, colourless
 0.30 × 0.24 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 1998)
 $T_{\min} = 0.823$, $T_{\max} = 1.00$
 10276 measured reflections
 3660 independent reflections
 2457 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$
 $\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.139$
 $S = 1.06$
 3660 reflections
 263 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2 + 0.4139P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

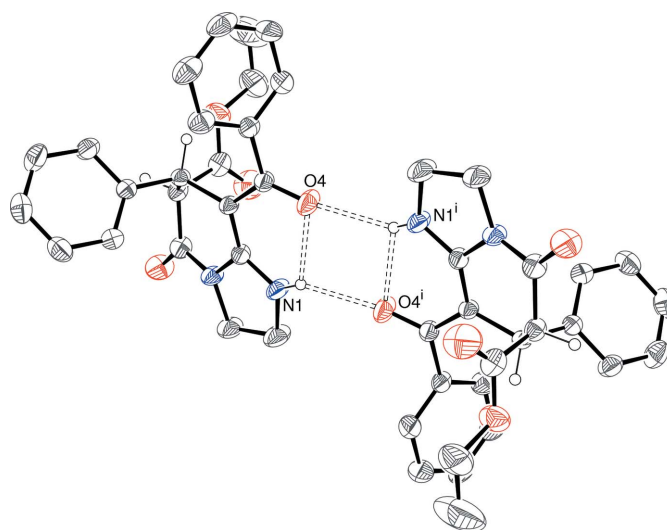


Figure 2
The dimeric association of molecules of (I). Dashed lines indicate hydrogen bonds. [Symmetry code: (i) 1 - x, 1 - y, -z.]

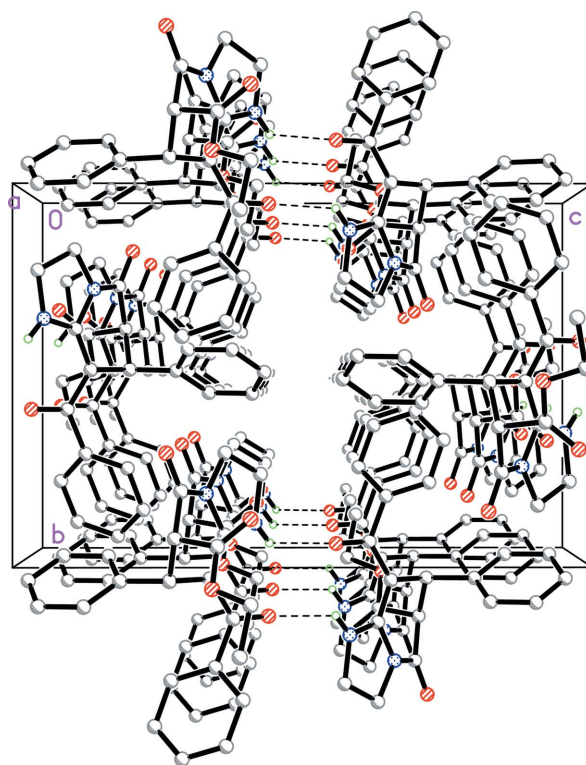


Figure 3
The packing in (I), viewed down [100], with hydrogen bonds indicated by dashed lines. All C-bound H atoms have been omitted for clarity.

Table 1

Selected bond lengths (Å).

C1–N1	1.329 (3)	C1–C16	1.378 (3)
C1–N2	1.390 (3)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O4$	0.86	2.20	2.720 (3)	119
$N1-H1\cdots O4^i$	0.86	2.19	2.854 (3)	134

Symmetry code: (i) $-x + 1, -y + 1, -z$.

All H atoms were placed in geometrically idealized positions ($C-H = 0.95-1.00$ Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$, or $U_{iso}(H) = 1.5U_{eq}(\text{methyl carrier})$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1998); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:

SHELXTL (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

Dr Hong-Geng Wang of Nankai University collected the intensity data.

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