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Ethyl 4-benzoyl-3,5-diphenylpyrrolidine-2-carboxylate

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Key indicators

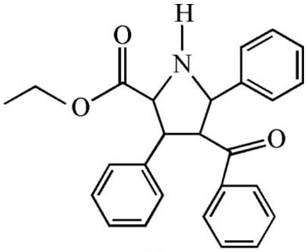
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.036
wR factor = 0.095
Data-to-parameter ratio = 8.1

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

The title compound, $C_{26}H_{25}NO_3$, was synthesized by the 1,3-dipolar cycloaddition reaction of benzaldehyde, α-bromoacetophenone and *N*-benzylideneglycine ethyl ester. In the molecule, the heterocyclic five-membered ring adopts an envelope conformation.

Comment

The 1,3-dipolar cycloaddition reaction is one of the most efficient and widely used methods for the synthesis of nitrogen-containing five-membered heterocycles (Grigg, 1995). The title compound, (I), is a new compound synthesized recently in our laboratory by an extension of the 1,3-dipolar cycloaddition reaction (Gu *et al.*, 2002) in one pot. We present its crystal structure here.



(I)

In (I) (Fig. 1), all bond lengths and angles are normal (Allen *et al.*, 1987). The central five-membered ring has an envelope conformation. Atoms N1, C8, C9 and C20 define the mean plane (A), with an r.m.s. deviation of 0.0457 (3) Å, and atom C16 is displaced by 0.5945 (3) Å as the flap of the envelope. The planes A and N1/C16/C20 make a dihedral angle of 40.5 (3)°. Phenyl ring C2–C7 makes dihedral angles of 75.9 (4) and 95.5 (4)° with phenyl rings C10–C15 and C21–C26, respectively. The crystal structure exhibits no classical hydrogen bonds.

Experimental

Benzaldehyde (3 mmol) and *N*-benzylideneglycine ethyl ester (3 mmol) were added to a solution of α-bromoacetophenone (1 mmol) in dichloromethane (15 ml). To the stirred mixture, zinc powder (2 mmol) was added. After the mixture had been refluxed for 16 h, the reaction was quenched with a saturated solution of ammonium chloride (10 ml). The mixture was extracted with diethyl ether, evaporated and separated by flash chromatograghy (ethyl acetate–petroleum ether). A colourless powder was obtained. Single crystals suitable for X-ray diffraction were obtained (yield 52%) by slow evaporation of an ethyl acetate–petroleum ether (1:5) solution.

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Crystal data

$C_{26}H_{25}NO_3$
$M_r = 399.47$
Orthorhombic, $Pna2_1$
$a = 22.112$ (5) Å
$b = 5.7505$ (12) Å
$c = 16.555$ (4) Å
$V = 2105.0$ (8) Å^3

$Z = 4$
$D_x = 1.260$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 294$ (2) K
Block, colourless
0.32 × 0.28 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.974$, $T_{\max} = 0.981$

11174 measured reflections
2237 independent reflections
1511 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.055$
$\theta_{\max} = 26.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.096$
$S = 1.00$
2237 reflections
276 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.1059P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.11$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}

The C-bound H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.98 Å. For CH and CH_2 groups, $U_{\rm iso}$(H) values were set equal to $1.2U_{\rm eq}$(carrier atom) and for the methyl groups they were set equal to $1.5U_{\rm eq}$(carrier atom). Atom H1*A* was located in a difference map and refined with a distance restraint of N—H = 0.91 (1) Å and with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(N). In the absence of significant anomalous scattering effects, 1884 Friedel pairs were merged.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

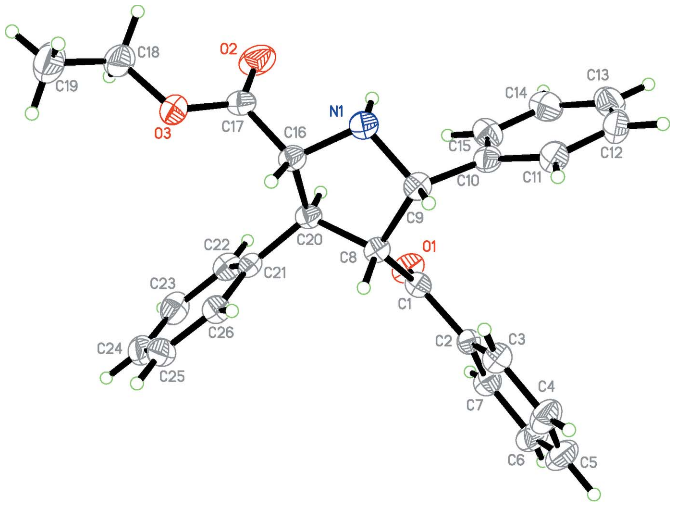

Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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