

Ethyl 4-oxo-2,6-diphenylpiperidine-3-carboxylate

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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
Disorder in main residue
 R factor = 0.069
 wR factor = 0.206
Data-to-parameter ratio = 13.5

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

In the title compound, $\text{C}_{20}\text{H}_{21}\text{NO}_3$, the piperidine ring adopts a chair conformation. The N atom of the piperidine ring shows pyramidal character.

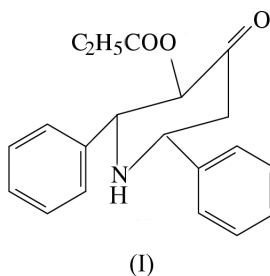
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Comment

The preferred conformation of the piperidine ring has been investigated by chemical and physical methods, and in most derivatives it is found to exist in a chair conformation (Ramalingam *et al.*, 1979). Also, investigations based on photoelectron spectroscopy and semi-empirical calculations show a pyramidal N atom for the piperidine ring (Woydt *et al.*, 1991). With a view to determining the conformational preference of the piperidine ring when it is substituted with bulky groups like the phenyl group and the ethoxycarbonyl group, the structure determination of the title compound, (I), was undertaken.



The piperidine ring of (I) adopts a slightly distorted chair conformation, with puckering amplitude $Q = 0.543$ (2) Å, $\theta = 13.9$ (2)° and $\varphi = 351.7$ (10)° (Cremer & Pople, 1975). The puckering is enhanced at N1 and decreased at C4 (Sekar, Parthasarathy & Rajalingam, 1990; Sekar, Parthasarathy & Radhakrishnan, 1990; Sekar *et al.*, 1993; Ianelli *et al.*, 1992; Kooijman *et al.*, 1997). The sum of bond angles around N1 (327.7°) indicates sp^3 hybridization for N1. The two phenyl rings are equatorially attached to the piperidine ring. The dihedral angle between the two phenyl ring planes is 37.9 (2)°.

Experimental

The title compound was synthesized by condensation of 9.90 g (0.100 mol) piperidin-4-one and 10.7 ml (0.100 mol) ethyl acetate in the presence of 0.05 g benzaldehyde and benzyl ketone. The reaction mixture was heated for about 16 h. The solvent was evaporated and the crystals were purified by recrystallization from methanol (m.p. 433 K, yield 70%).

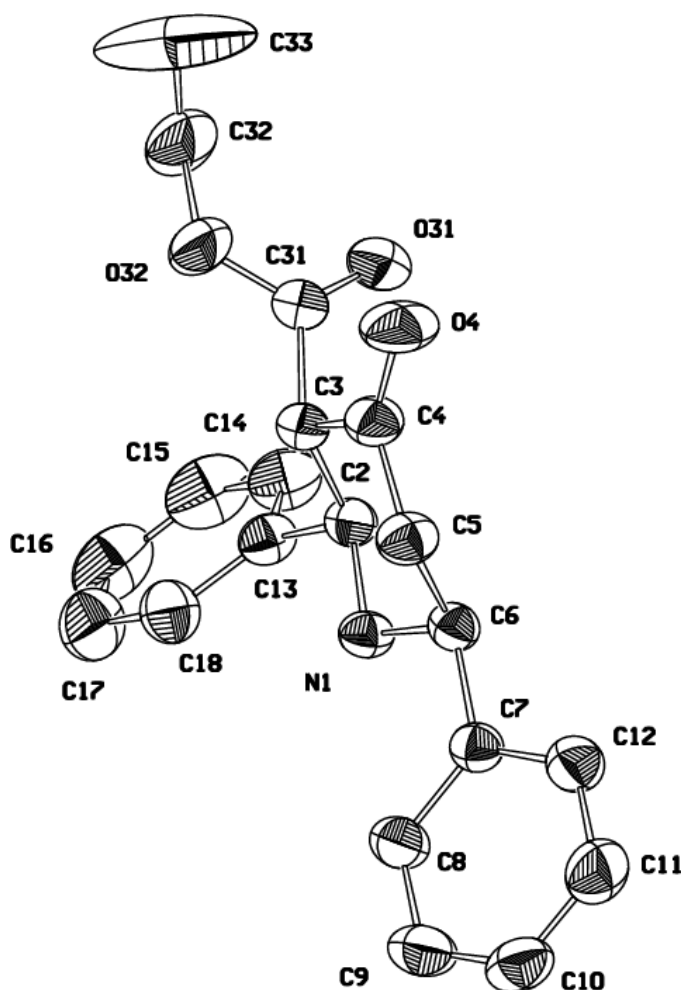


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids. For clarity, only the major (55%) conformer of the ethoxycarbonyl group is shown. All H atoms have been omitted.

Crystal data

$C_{20}H_{21}NO_3$
 $M_r = 323.38$
 Monoclinic, $P2_1/c$
 $a = 10.124$ (2) Å
 $b = 7.801$ (3) Å
 $c = 22.896$ (3) Å
 $\beta = 100.39$ (1)°
 $V = 1778.6$ (8) Å³
 $Z = 4$

Data collection

Enraf–Nonius CAD-4
 diffractometer
 ω -2 θ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\min} = 0.972$, $T_{\max} = 0.986$
 3430 measured reflections
 3236 independent reflections
 2629 reflections with $I > 2\sigma(I)$

$D_x = 1.208$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25
 reflections
 $\theta = 2$ –25°
 $\mu = 0.08$ mm⁻¹
 $T = 293$ (2) K
 Needle, colourless
 0.30 × 0.17 × 0.09 mm

$R_{\text{int}} = 0.047$
 $\theta_{\text{max}} = 25.3^\circ$
 $h = 0 \rightarrow 12$
 $k = 0 \rightarrow 9$
 $l = -27 \rightarrow 27$
 2 standard reflections
 every 60 reflections
 intensity decay: negligible

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.069$
 $wR(F^2) = 0.206$
 $S = 1.10$
 3236 reflections
 240 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.1093P)^2 + 0.5001P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.44$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.41$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.011 (1)

Table 1

Selected torsion angles (°).

| | | | |
|--------------|-------------|-------------|--------------|
| C6–N1–C2–C13 | 175.15 (17) | C3–C4–C5–C6 | 45.1 (3) |
| C6–N1–C2–C3 | –63.5 (2) | C2–N1–C6–C5 | 64.8 (2) |
| N1–C2–C3–C4 | 49.5 (2) | C4–C5–C6–N1 | –53.3 (2) |
| C2–C3–C4–C5 | –42.9 (3) | C4–C5–C6–C7 | –175.37 (18) |

Atoms C32 and C33 of the ethoxycarbonyl group were found to be disordered. The occupancies of the disordered positions C32/C32' and C33/C33' were initially refined and later fixed at 55/45%. The corresponding O–C and C–C distances in the major and minor conformers were restrained to be equal. The H atom attached to N1 was located from a difference map and isotropically refined. All other H atoms were geometrically positioned and allowed to ride on their parent atoms.

Data collection: *CAD-4 Software* (Frenz, 1989); cell refinement: *MolEN* (Fair, 1990); data reduction: *MolEN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP97* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL97*.

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