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Methyl 4-oxo-*r*-2,*c*-6-diphenylpiperidine-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.136
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.

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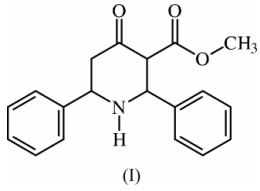
The crystal structure of the title compound, $C_{19}H_{19}NO_3$, was determined because it is believed to possess medicinal properties. The piperidine ring assumes a chair conformation. The benzene-ring and methoxycarbonyl substituents are oriented equatorially. Whereas C—H···O intermolecular hydrogen bonds stabilize the crystal packing, the NH group does not form any hydrogen bond.

Comment

Piperidine derivatives are used clinically to prevent post-operative vomiting, to speed up gastric emptying before anaesthesia, to facilitate radiological investigations and to correct a variety of disturbances of gastrointestinal functions (Robinson, 1973). Several 2,6-disubstituted piperidines are found to be useful as tranquilisers and some possess hypotensive activity (Kumar *et al.*, 1998), and a combination of stimulant and depressant effects on the central nervous system (Ganellin & Spickett, 1965), as well as bactericidal, fungicidal and herbicidal activities.



(I)

A *ZORTEP* plot (Zsolnai, 1998) of the title molecule, (I), is shown in Fig. 1. The piperidine ring adopts a chair conformation. Both benzene rings are attached to it in equatorial

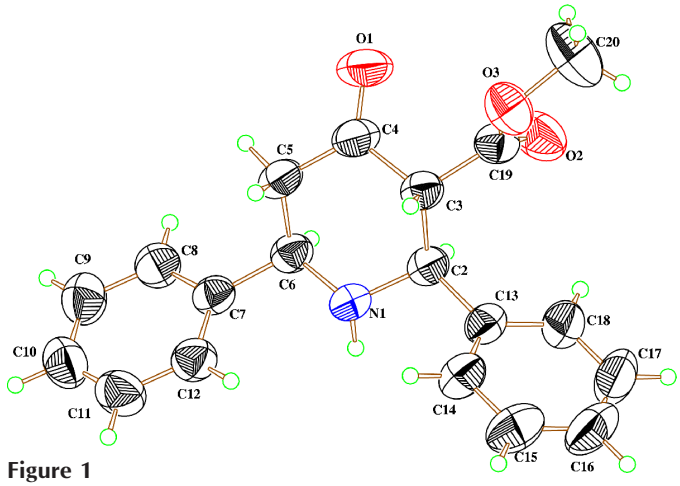


Figure 1
ZORTEP plot (Zsolnai, 1998) of the title molecule showing 50% probability displacement ellipsoids.

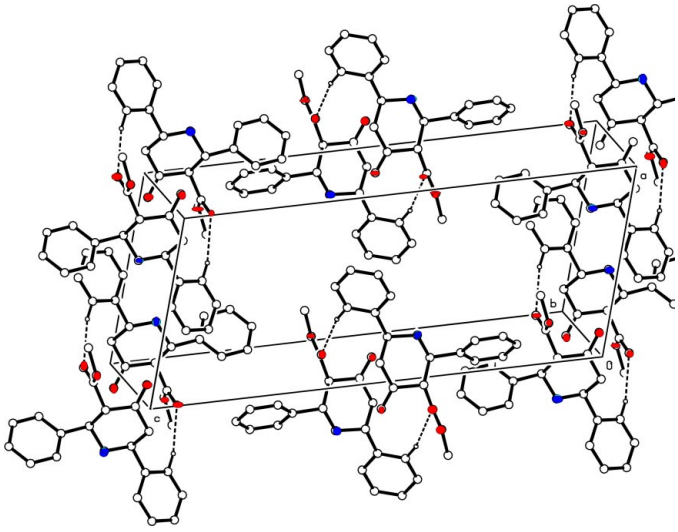


Figure 2
The packing of the title compound. The dashed lines show the hydrogen bonding.

positions. The dihedral angle subtended between the planes of the benzene rings is 32.1 (4)°. The methoxycarbonyl group is attached to the piperidine ring in an equatorial position.

The packing of the molecules is shown in Fig. 2. C—H···O intermolecular hydrogen bonds stabilize the molecules in the crystal structure. The NH group does not form a hydrogen bond because it is shielded by the two adjacent benzene rings.

Experimental

The title compound was synthesized by a Mannich condensation reaction using benzaldehyde, methyl acetoacetate and ammonium acetate in a 2:1:1 ratio (Noller & Baliah, 1948) in 99% ethyl alcohol solution, refluxed for 1 h and then kept overnight. The colourless crystals obtained were recrystallized from ethanol by slow evaporation.

Crystal data

$C_{19}H_{19}NO_3$
M_r = 309.35
Monoclinic, $P2_1/c$
a = 9.7563 (13) Å
b = 7.710 (1) Å
c = 22.810 (3) Å
β = 102.348 (4)°
V = 1676.1 (4) Å^3
Z = 4
D_x = 1.226 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3296 reflections
θ = 1.8–26.0°
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, pale yellow
0.41 × 0.20 × 0.12 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
12 559 measured reflections
3296 independent reflections
2595 reflections with $I > 2\sigma(I)$
R_{int} = 0.077
θ_{max} = 26.0°
$h = -12 \rightarrow 12$
$k = -8 \rightarrow 9$
$l = -28 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.136
S = 1.05
3296 reflections
213 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0673P)^2 + 0.137P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.015 (2)

Table 1
Selected geometric parameters (Å, °).

C4—O1	1.2030 (17)	C19—O3	1.3321 (17)
C19—O2	1.1965 (17)		
C2—N1—C6—C7	176.64 (10)	C13—C2—C3—C4	−171.38 (11)
C2—N1—C6—C5	−63.66 (14)	C19—C3—C4—C5	169.90 (12)
C6—N1—C2—C13	−175.53 (10)	C2—C3—C4—C5	45.82 (16)
C6—N1—C2—C3	63.25 (14)	C3—C4—C5—C6	−47.47 (17)
N1—C2—C3—C19	−172.71 (10)	N1—C6—C5—C4	53.33 (15)
N1—C2—C3—C4	−51.02 (14)	C7—C6—C5—C4	174.75 (11)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O2^{i}	0.930	2.49	3.386 (2)	163

Symmetry code: (i) $2-x, -y, 1-z$.

The H atom bonded to nitrogen was refined isotropically. H atoms bonded to carbon were positioned geometrically and refined using a riding model [C—H = 0.93–0.98 Å, and U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(C_{methyl})].

Data collection: *SMART* (Siemens, 2000); cell refinement: *SAINT* (Siemens, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1998) and *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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