Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2,6-Diphenyl-3-isopropylpiperidin-4-one

M. Nilofar Nissa,[a] D. Velmurugan,[a]* S. Narasimhan,[b] V. Rajagopal[b] and Moon-Jib Kim[c]

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [b]Spic Science Foundation, Guindy, Chennai 600 035, India, and [c]Department of Physics, Soonchunhyang University, PO Box, Asan, Chungnam 336-600, Korea

Correspondence e-mail: d_velu@yahoo.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.063
wR factor = 0.195
Data-to-parameter ratio = 14.4

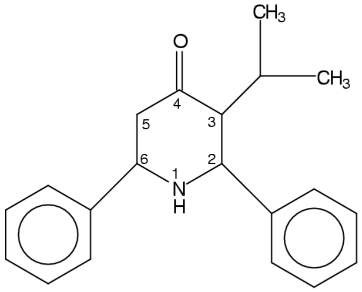
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 17 September 2001
Accepted 27 September 2001
Online 6 October 2001

In the title compound, the two phenyl rings are oriented at an angle of 63.4 (1)° with respect to each other. The 4-piperidone ring adopts a slightly distorted chair conformation. Molecules are packed through N—H···O hydrogen bond and van der Waals interactions.

Comment

The piperidine ring is a characteristic feature of antihistaminic agents, oral anesthetics, narcotic analgesics, tranquillizers and hypotensive agents (Robinson, 1973). Many piperidine derivatives also form the skeleton of several alkaloids (Hootele *et al.*, 1980). Several 2,6-substituted piperidine derivatives are found to possess fungicidal, herbicidal and bactericidal activities (Mobio *et al.*, 1989). A large variety of 4-piperidones with different substituents in the 1, 2, 3, 5 and 6 positions with further substitutions in the 2- and 6-substituent phenyl rings are reported elsewhere (Jia *et al.*, 1989*a*,*b*; Cheer *et al.*, 1984; Sekar *et al.*, 1990, 1993; Sukumar *et al.*, 1994; Díaz *et al.*, 1997). Continuing this series, we report here the structure of 2,6-diphenyl-3-isopropylpiperidin-4-one, (I). Colourless prism-shaped crystals obtained during the synthesis were suitable for X-ray crystallography.



(I)

Fig. 1 shows the molecular structure; bond lengths and angles are given in Table 1. The piperidine ring adopts a slightly distorted chair conformation. Atoms C2, C3, C5 and C6 lie in a plane, whereas N1 and C4 deviate by −0.703 (3) and 0.518 (4) Å on either side of this plane. The phenyl rings are planar, with a maximum deviation of −0.009 (4) Å for ring *A* (C7—C8) and 0.003 (5) Å for ring *B* (C13—C14), and are *cis* to the piperidine ring [C5—C6—C7—C8 = 59.0 (5)°; C3—C2—C13—C14 = −72.5 (4)°]. The dihedral angle between the two phenyl rings is 63.4 (1)°.

The bond lengths of the rings vary from 1.352 (5) to 1.386 (5) Å. The bond lengths C6—C7 [1.513 (5) Å] and C2—C13 [1.503 (5) Å] are in good agreement with the overall distances of Csp^3—C$_{ar}$ [1.513 (14) Å] (Allen *et al.*, 1987). The endocyclic angles C7—C8—C12 [117.3 (3)°] and C13—C14—C18 [117.8 (3)°] are smaller than the average value of 120° as

DOI: 10.1107/S1600536801016026

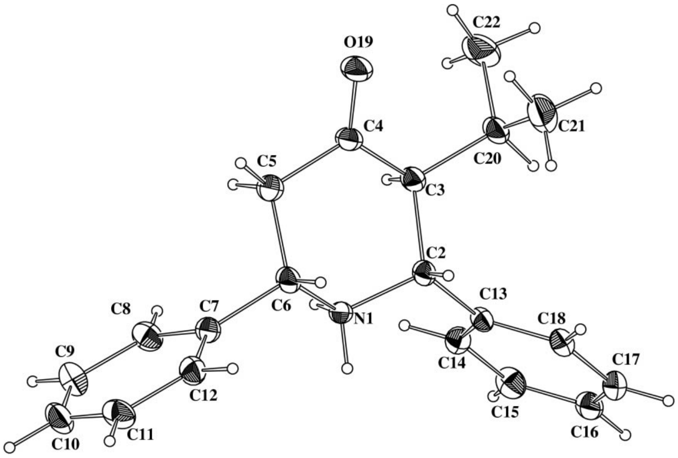


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme and 50% probability level displacement ellipsoids for the non-H atoms.

observed in related structures (Singh *et al.*, 1990; Eswaramoorthy, 1992). This may be due to conjugation.

Molecules are packed in chains running along the *b* axis. These continuous chains are stabilized by N—H···O intermolecular hydrogen bonds (Table 2). Further stabilization is provided by the shortest C—H···O contact and by van der Waals interactions.

Experimental

The procedure adopted for the preparation of the title heterocyclic compound is similar to that of Noller & Baliah (1948). 0.2 mol of ammonium acetate was dissolved in 30 ml of rectified spirit, and 0.4 mol of benzaldehyde and 0.2 mol of isobutylmethyl ketone were added to this solution. The mixture was heated to boiling point and set aside for a day. The oily base obtained was converted into its hydrochloride by the addition of concentrated hydrochloric acid and the separated solid was filtered, dried and recrystallized from absolute ethanol. To the hydrochloride, a few drops of acetone were added and the mixture was neutralized with liquid ammonia. On dilution with water, the free base was separated and was filtered and recrystallized from absolute ethanol; m.p. 398–399 K.

Crystal data

$C_{20}H_{23}NO$
$M_r = 293.39$
Monoclinic, $P2_1/c$
$a = 11.6008\ (8)$ Å
$b = 13.459\ (3)$ Å
$c = 12.0597\ (12)$ Å
$\beta = 118.611\ (9)^\circ$
$V = 1653.0\ (5)$ Å^3
$Z = 4$
$D_x = 1.179$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 6.2$–11.6°
$\mu = 0.07$ mm^{-1}
$T = 293\ (2)$ K
Prism, colourless
0.4 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
3046 measured reflections
2897 independent reflections
1320 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\max} = 25.0^\circ$
$h = -13 \rightarrow 12$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 14$
3 standard reflections every 100 reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.195$
$S = 0.96$
2897 reflections
201 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.061$
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C6	1.447 (4)	C4—O19	1.204 (4)
N1—C2	1.463 (4)	C4—C5	1.498 (5)
C2—C3	1.550 (5)	C5—C6	1.520 (5)
C3—C4	1.504 (5)	C20—C22	1.518 (6)
C3—C20	1.537 (5)	C20—C21	1.499 (6)
C6—N1—C2	112.4 (3)	O19—C4—C5	120.2 (3)
N1—C2—C13	110.7 (3)	C3—C4—C5	116.8 (3)
N1—C2—C3	108.5 (3)	N1—C6—C7	112.6 (3)
C13—C2—C3	112.8 (3)	N1—C6—C5	108.0 (3)
C4—C3—C2	109.6 (3)	C7—C6—C5	111.8 (3)
C4—C3—C20	115.2 (3)	C22—C20—C21	112.2 (4)
C2—C3—C20	114.6 (3)	C22—C20—C3	110.3 (3)
O19—C4—C3	122.8 (3)	C21—C20—C3	114.8 (3)
C6—N1—C2—C3	−66.4 (4)	C2—N1—C6—C5	65.5 (4)
N1—C2—C3—C4	52.3 (4)	C4—C5—C6—N1	−52.0 (4)
C13—C2—C3—C20	−53.5 (4)	N1—C6—C7—C8	−62.9 (5)
C20—C3—C4—O19	9.6 (6)	C4—C3—C20—C22	−70.2 (5)
C2—C3—C4—C5	−44.3 (5)	C2—C3—C20—C22	161.3 (3)
C3—C4—C5—C6	44.8 (5)	C4—C3—C20—C21	57.7 (5)
C2—N1—C6—C7	−170.6 (3)	C2—C3—C20—C21	−70.8 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O19$^{\rm i}$	0.90	2.22	3.101 (4)	166
C9—H9···O19$^{\rm ii}$	0.93	2.80	3.584 (6)	143

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $1 - x, -y, 2 - z$.

All H atoms were fixed geometrically and refined isotropically using a riding model with N—H = 0.86, C—H(aromatic) = 0.93 and C—H(aliphatic) = 0.98 Å.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Enraf–Nonius, 1994); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLUTON* (Spek, 1997); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1983, 1995).

MN thanks the CSIR for a Senior Research Fellowship.

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.
Cheer, C. J., Cosgrove, J. P. & Vittimberga, B. M. (1984). *Acta Cryst.* C**40**, 1474–1475.
Díaz, E., Barrios, H. & Toscano, R. A. (1997). *Acta Cryst.* C**53**, 1468–1471.
Enraf–Nonius (1994). *CAD*-4 *EXPRESS* and *XCAD*4. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.
Eswaramoorthy, S. (1992). PhD Thesis, University of Madras, Chennai 600 025, India.

Hootele, C., Colan, B. & Halin, F. F. (1980). *Tetrahedron Lett.* **21**, 5063–5066.

Jia, Z., Quail, J. W., Arora, V. K. & Dimmock, J. R. (1989*a*). *Acta Cryst.* C**45**, 285–289.

Jia, Z., Quail, J. W., Arora, V. K. & Dimmock, J. R. (1989*b*). *Acta Cryst.* C**45**, 1117–1118.

Mobio, I. G., Soldatenkov, A. T., Federov, V. O., Ageev, E. A., Sargeeva, N. D., Lin, S., Stashenko, E. E., Prostakov, N. S. & Andreeva, E. I. (1989). *Khim. Farm. Zh.* **23**, 421–427.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Noller, C. R. & Baliah, V. (1948). *J. Am. Chem. Soc.* **70**, 3853–3855.

Robinson, O. P. W. (1973). *Postgrad. Med. J. (Suppl.),* **49**, 9–12.

Sekar, K., Parthasarathy, S. & Radhakrishnan, T. R. (1990). *Acta Cryst.* C**46**, 1338–1340.

Sekar, K., Parthasarathy, S. & Radhakrishnan, T. R. (1993). *Acta Cryst.* C**49**, 93–95.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Singh, P., Levine, S. G. & Kasdorf, K. (1990). *Acta Cryst.* C**46**, 2469–2470.

Spek, A. L. (1997). *PLUTON*. University of Utrecht. The Netherlands.

Sukumar, N., Ponnuswamy, M. N., Thenmozhiyal, J. C. & Jeyaraman, R. (1994). *Bull. Chem. Soc. Jpn*, **67**, 1–5.

Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.