Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,6-Bis(4-methylphenyl)-3-phenylpiperidin-4-one

M. Subha Nandhini,[a] M. Srinivasan,[b] R. V. Krishnakumar,[c] A. Mostad,[d] S. Perumal,[b] S. Selvaraj[b] and S. Natarajan[a]*

[a]Department of Physics, Madurai Kamaraj University, Madurai 625021, India, [b]Department of Chemistry, Madurai Kamaraj University, Madurai 625021, India, [c]Department of Physics, Thiagarajar College, Madurai 625009, India, and [d]Department of Chemistry, University of Oslo, PO Box 1033 Blindern, N-0315 Oslo 3, Norway

Correspondence e-mail: s_natarajan50@yahoo.com

Key indicators

Single-crystal X-ray study
T = 105 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.127
Data-to-parameter ratio = 16.9

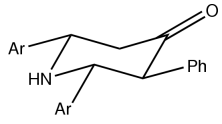
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 27 August 2003
Accepted 9 September 2003
Online 24 September 2003

In the title molecule, $C_{25}H_{25}NO$, the piperidone ring adopts a slightly distorted chair conformation. The two methylphenyl rings are oriented differently with reference to the central piperidone plane. The crystal packing is characterized by C—H···π and C—H···O interactions. It can be concluded that the stable conformation is the same in the solid state as in solution.

Comment

2,6-Disubstituted piperidine derivatives are found to possess fungicidal, bactericidal and herbicidal activities (Mobio *et al.*, 1989). A series of investigations on the synthesis and NMR studies of 2,6-disubstituted piperidine derivatives with variation in the substituent groups and exchange of their position in the phenyl rings is underway in our laboratory. It is known that the influence of a simple exchange of substituents in organic molecules leads to totally different crystal and molecular structures. In this context, elucidation of crystal structures of these molecules using precise X-ray data are of considerable interest and importance, and are expected to throw light on the nature of specific interactions and consequent structure–function relationships. We report here the structure of 2,6-bis(4-methylphenyl)-3-phenylpiperidin-4-one, (I).



Ar = 4-MeC$_6$H$_4$

(I)

Fig. 1 shows the atom-numbering scheme adopted for (I). The piperidone ring has a slightly distorted chair conformation, which seems to be similar to those observed in related structures reported by Nilofar Nissa *et al.* (2001), Singh *et al.* (1990), Sekar *et al.* (1990, 1993) and Jia *et al.* (1988). Also, NMR studies (Perumal *et al.*, 2003) are found to agree well with the conformation found in the solid state. The *p*-methylphenyl and phenyl rings are oriented equatorially with respect to the piperidone ring.

The C—C and C—N distances agree well with expected values. The angle at which the 4-methyphenyl at C6 [70.1 (1)°] is oriented with reference to the plane constituting atoms C2, C3, C5 and C6 of the piperidone ring is less than that of the 4-methylphenyl at C2 [87 (1)°] and 3-phenyl at C3 [81.9 (1)°]. This can be ascribed to the greater steric interaction between the 4-methylphenyl group at C2 and the phenyl ring at C3.

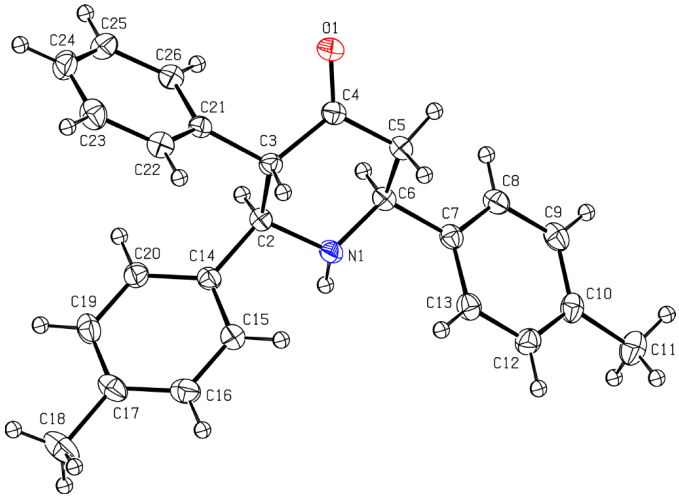


Figure 1
Molecular structure of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids.

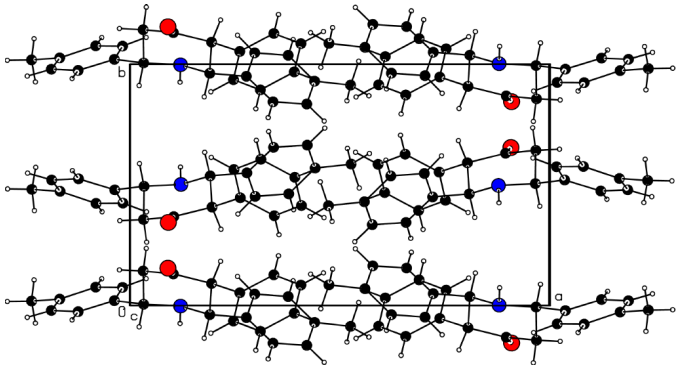


Figure 2
Packing diagram of (I), viewed down the *c* axis.

While the the 4-methylphenyl ring at C6 does not have much steric interactions with the groups present in the piperidone ring system. It is also pertinent to note that the torsion angle H2—C2—C3—H3 of 172° points to a larger deviation (8°) from the perfectly diaxial (*anti*) relationship, while the torsion angle H6—C6—C5—H5*A* (in sterically less crowded positions) of −177°, shows a smaller deviation (3°) from the perfectly diaxial relationship.

In the crystal structure, inversion-related molecules aggregate into layers parallel to the *ac* plane (Fig. 2). Adjacent layers are linked together by C—H···π interactions (Table 2). The H atom, H1N, is not involved in any significant hydrogen bonds. Apart from the interactions listed in Table 2, there are no other notable intermolecular contacts.

Experimental

Ammonium acetate (0.95 g, 0.015 mol) was dissolved in ethanol (3 ml) by heating. 4-Methylbenzaldehyde (1.8 ml, 0.015 mol) and phenylacetone (1 g, 0.0075 mol) were added to this solution, and the mixture was heated until the colour of the solution changed to orange. The solution was stored at room temperature for 2 days, the precipitate filtered off, washed with cold ethanol and recrystallized from ethanol–ethyl acetate (4:1) mixture; yield: 57%.

Crystal data

$C_{25}H_{25}NO$
M_r = 355.46
Monoclinic, $P2_1/c$
a = 15.214 (3) Å
b = 8.7408 (17) Å
c = 14.885 (3) Å
β = 90.75 (3)°
V = 1979.3 (7) Å^3
Z = 4
D_x = 1.193 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1024 reflections
θ = 1.3–26.8°
μ = 0.07 mm^{-1}
T = 105 (2) K
Prism, colourless
0.3 × 0.3 × 0.2 mm

Data collection

Bruker SMART area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.98, T_{max} = 0.99
22265 measured reflections
4231 independent reflections
3499 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 26.8°
$h = -19 \rightarrow 19$
$k = -11 \rightarrow 11$
$l = -18 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.128
S = 1.06
4231 reflections
250 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.071P)^2 + 0.5878P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.2120 (16)	C3—C4	1.5306 (18)
N1—C2	1.4675 (17)	C4—C5	1.5075 (18)
N1—C6	1.4689 (17)	C5—C6	1.5398 (18)
C2—C3	1.5586 (18)		
C6—N1—C2—C14	177.47 (10)	C2—C3—C4—C5	−43.50 (15)
C6—N1—C2—C3	−61.87 (14)	C3—C4—C5—C6	47.79 (15)
N1—C2—C3—C21	176.25 (10)	C2—N1—C6—C5	65.86 (14)
N1—C2—C3—C4	48.01 (14)	C4—C5—C6—N1	−56.19 (14)
C2—C3—C4—O1	138.49 (13)	C4—C5—C6—C7	−177.50 (10)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O1^{i}	0.93	2.67	3.507 (2)	150
C6—H6···O1^{i}	0.98	2.67	3.407 (2)	132
C2—H2···*Cg*1^{ii}	0.98	3.22	4.051 (2)	143
C5—H5A···*Cg*1^{iii}	0.97	2.70	3.573 (2)	150
C25—H25···*Cg*2^{iv}	0.93	2.83	3.662 (2)	150
C20—H20···*Cg*3	0.93	3.36	3.702 (2)	104

Symmetry codes: (i) $-x, 2-y, 1-z$; (ii) $-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iii) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iv) $x, \frac{3}{2}-y, \frac{1}{2}+z$. Notes: *Cg*1, *Cg*2 and *Cg*3 denote the centroids of rings C7–C10/C12,C13, C14–C17/C19,C20 and C21–C26, respectively.

Atom H1N was found in a difference Fourier map and its positional parameters and U_{iso} value were refined. All other H atoms were placed in calculated positions and allowed to ride on their parent C atoms, with C—H distances in the range 0.93–0.98 Å and the U_{iso} values set to $1.5U_{eq}$(parent atom) for the methyl H atoms and $1.2U_{eq}$(parent atom) for the other H atoms. A rotating-group model was used for the methyl groups.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL*97.

MSN and SN thank the UGC for the Special Assistance Programme. SP thanks the UGC and DST, India, for major Research Projects.

References

Bruker (1998). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SMART-NT* (Version 5.0) and *SAINT-NT* (Version 5.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Jia, Z.-C., Quail, J. W., Arora, V. K. & Dimmock, J. R. (1988). *Acta Cryst.* C**44**, 2114–2117.
Mobio, I. G., Soldatenkov, A. T., Federov, V. O., Ageev, E. A., Sargeeva, N. D., Lin, S., Stashenko, E. E., Prostakov, N. S. & Andreeva, W. I. (1989). *Khim. Farm. Zh.* **23**, 421–427.
Nilofar Nissa, M., Velmurugan, D., Narasimhan, S., Rajagopal, V. & Kim, M.-J. (2001). *Acta Cryst.* E**57**, o996–o998.
Perumal, S., Srinivasan, M. & Selvaraj, S. (2003). *Indian J. Chem.* Submitted.
Sekar, K., Parthasarathy, S. & Radhakrishnan, T. R. (1990). *Acta Cryst.* C**46**, 1153–1155.
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. & Kasdore, K. (1990). *Acta Cryst.* C**46**, 2469–2470.
Spek, A. L. (1999). *PLATON*. Utrecht University, The Netherlands.