Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,6-Diphenyl-1-propylpiperidine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.053
wR factor = 0.169
Data-to-parameter ratio = 18.4

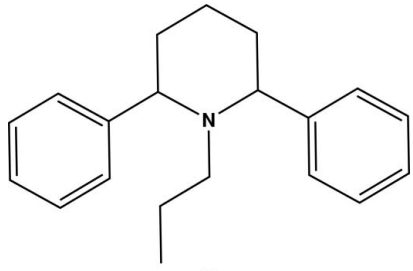
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 18 August 2006
Accepted 23 August 2006

The title molecule, $C_{20}H_{25}N$, possesses crystallographic mirror symmetry. The piperidine ring adopts a chair conformation. The crystal packing is stabilized by weak C—H···π intermolecular interactions, which form a chain along the *b* axis.

Comment

Piperidine, one of the simplest heterocyclic systems, is found in nature as a part of several alkaloid compounds. It is a characteristic feature of antihistaminic agents, anesthetics, tranquilizers and hypotensive agents (Robinson, 1973). Both natural and synthetic piperidine derivatives have high pharmaceutical value. Several 2,6-disubstituted piperidine derivatives have fungicidal, herbicidal and bactericidal properties (Mobio *et al.*, 1989).



(I)

The title molecule, (I), possesses crystallographically imposed mirror symmetry, with atoms C3, N1, C10, C11, C12 and H12*A* located on the mirror plane (Fig. 1). The bond lengths and angles are comparable with literature values (Allen *et al.*, 1987). The sum of the bond angles around atom N1 (332.5°) indicates sp^3 hybridization. The piperidine ring adopts a chair conformation. The puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) are q_2 = 0.018 (1), q_3 = 0.558 (1), Q_T = 0.559 (1) Å and φ = 2.1 (1)°. The dihedral angle between the two symmetry-related phenyl rings is 55.52 (4)°.

The crystal packing is stabilized by weak C—H···π intermolecular interactions. Atom C2 at (*x*, *y*, *z*) acts as a donor to the C4–C9 phenyl ring (centroid *Cg*1) of a centrosymmetrically related molecule at (1 − *x*, 1 − *y*, −*z*) through H2*A*, generating a dimer with an H···*Cg*1 separation of 3.04 Å. The C—H···π dimers form a chain along the *b* axis (Fig. 2).

Experimental

To a homogenous solution of 1,3-diphenylpropane (1.19 mmol, 300 mg, 1 equivalent), propylamine (11.9 mmol, 700 mg, 10 equivalents) and polyethyleneglycol-200 (10 ml) in a 25 ml Erlenmeyer flask, 85% formic acid (11.9 mmol, 700 mg, 1 ml, 10 equivalents) was added at 273–278 K. The reaction mixture was then irradiated in a

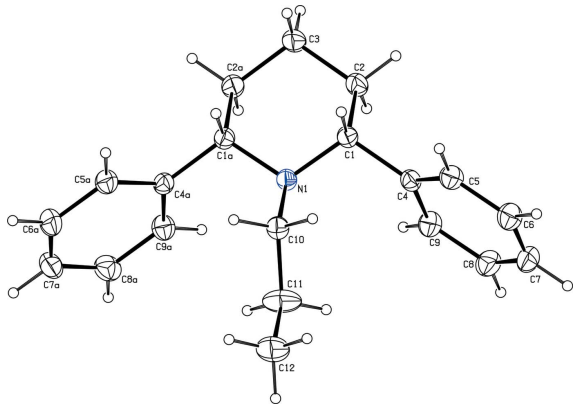


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids. Atoms labelled with the suffix a are generated by the symmetry operation $(x, \frac{1}{2} - y, z)$.

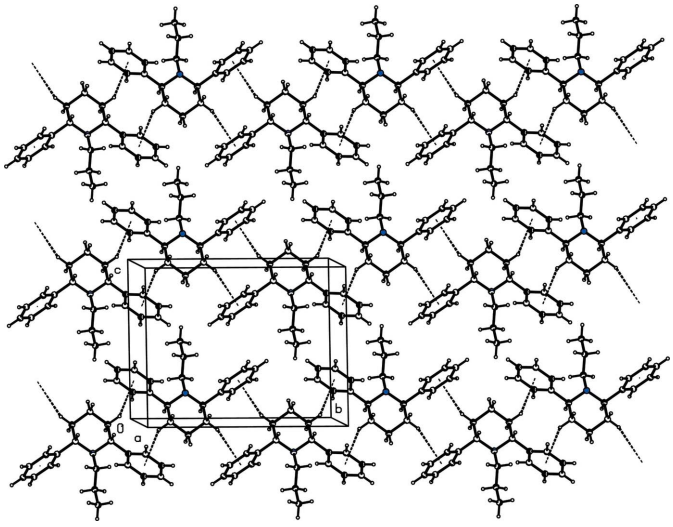


Figure 2
The crystal packing of (I), viewed down the *a* axis. C—H···π interactions are shown as dashed lines.

domestic microwave oven for 3 min at 370 W by which time dibenzoylpropane was absent (thin-layer chromatography). After completion of the reaction, the reaction mixture was added to ice-cold water (25 ml) and the pH of the aqueous solution was adjusted to 11 with 1 N NaOH. The organic compounds were extracted with dichloromethane (DCM; 3 × 15 ml). The DCM solution was dried over over anhydrous Na_2SO_4 and evaporated under reduced pressure. The crude product was subjected to column chromatography on basic alumina by eluting with increasing amounts of ethyl acetate in hexanes (2% to 10%). After purification, compound (I) was obtained as a white crystalline solid (yield 70%, 223 mg). Single crystals of (I) were obtained by recrystallization from about 5% DCM in hexane.

Crystal data

$C_{20}H_{25}N$
M_r = 279.41
Monoclinic, $P2_1/m$
a = 5.6325 (4) Å
b = 13.8274 (11) Å
c = 10.7341 (8) Å
β = 99.105 (1)°
V = 825.47 (11) Å^3
Z = 2
D_x = 1.124 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.06 mm^{-1}
T = 293 (2) K
Block, colourless
0.25 × 0.22 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
9508 measured reflections
2026 independent reflections
1748 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.169
S = 1.00
2026 reflections
110 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1072P)^2 + 0.1196P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.474 (1)	C6—C7	1.363 (2)
C1—C4	1.511 (2)	C7—C8	1.379 (2)
C1—C2	1.525 (2)	C8—C9	1.380 (2)
C2—C3	1.513 (2)	C10—N1	1.478 (2)
C4—C9	1.384 (2)	C10—C11	1.486 (3)
C4—C5	1.381 (2)	C11—C12	1.502 (3)
C5—C6	1.379 (2)		
C1^i—N1—C1	112.1 (1)	C1—N1—C10	110.2 (1)
C1^i—N1—C10	110.2 (1)		
N1—C1—C4—C9	58.3 (1)		

Symmetry code: (i) $x, -y + \frac{1}{2}, z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···$Cg1^{ii}$	0.97	3.04	3.800 (1)	136

Symmetry code: (ii) $-x + 1, -y + 1, -z$. $Cg1$ is the centroid of the C4–C9 phenyl ring.

Atoms H12*A* and H12*B* were located in a difference map and refined freely. The remaining H atoms were positioned geometrically and allowed to ride on their parent C atoms, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

Financial support from the University Grants Commission (UGC-SAP) and Department of Science and Technology (DST–FIST), Government of India, is acknowledged by DG and DV for providing facilities to the department.

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