

2-(2,6-Diphenylpiperidin-1-yl)ethanol

D. Gayathri,^a D. Velmurugan,^{a*}
K. Ravikumar,^b E. Poonguzhali^c
and H. Surya Prakash Rao^c^aDepartment of Crystallography and Biophysics,
University of Madras, Guindy Campus, Chennai
600 025, India, ^bLaboratory of X-ray
Crystallography, Indian Institute of Chemical
Technology, Hyderabad 500 007, India, and
^cDepartment of Chemistry, Pondicherry
University, Pondicherry 605 014, India

Correspondence e-mail: d_velu@yahoo.com

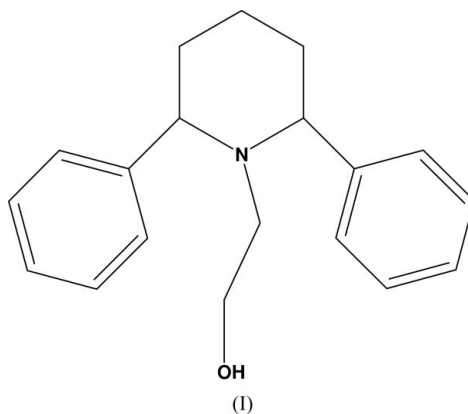
Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.056
 wR factor = 0.173
Data-to-parameter ratio = 19.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{19}\text{H}_{23}\text{NO}$, crystallizes with two molecules in the asymmetric unit. The piperidine ring adopts a chair conformation in both molecules. Intramolecular $\text{O}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\pi$ interactions are observed in only one molecule in the asymmetric unit. The crystal packing is stabilized by $\text{O}-\text{H}\cdots\text{O}$ intermolecular interactions, generating $R_4^4(8)$ rings.

Comment

The strong base, piperidine, is widely used as a building-block molecule in several industries. It is used as an intermediate in agrochemicals, pharmaceuticals and rubber vulcanization accelerators. Several 2,6-substituted piperidine derivatives possess fungicidal, herbicidal and bactericidal properties (Mobio *et al.*, 1989). In view of the above importance, the X-ray crystal structure determination of the title compound, (I), has been undertaken.



Compound (I) crystallizes in the triclinic system with two molecules, *A* and *B*, in the asymmetric unit. The bond lengths and angles are comparable with the corresponding values in a related structure which we published recently (Gayathri *et al.*, 2006). The sums of the bond angles around atom N1 [335.9 and 332.1° for molecules *A* and *B*] indicate sp^3 hybridization. The $\text{C}1-\text{N}1-\text{C}6-\text{C}7$, $\text{C}5-\text{N}1-\text{C}6-\text{C}7$ and $\text{N}1-\text{C}6-\text{C}7-\text{O}1$ torsion angles are 58.5 (2), -68.1 (2) and 174.1 (1)°, respectively, in molecule *A*, and -139.7 (1), 95.9 (2) and 50.7 (2)°, respectively, in molecule *B*.

The piperidine ring adopts a chair conformation in both molecules. The puckering parameters (Cremer & Pople, 1975) are $q_2 = 0.009$ (2) Å, $q_3 = 0.571$ (2) Å, $Q_T = 0.571$ (2) Å and $\varphi = 2.1$ (2)°, respectively, for molecule *A*, and $q_2 = 0.024$ (2) Å, $q_3 = 0.565$ (2) Å, $Q_T = 0.566$ (2) Å and $\varphi = 2.4$ (2)°, respectively, for molecule *B*.

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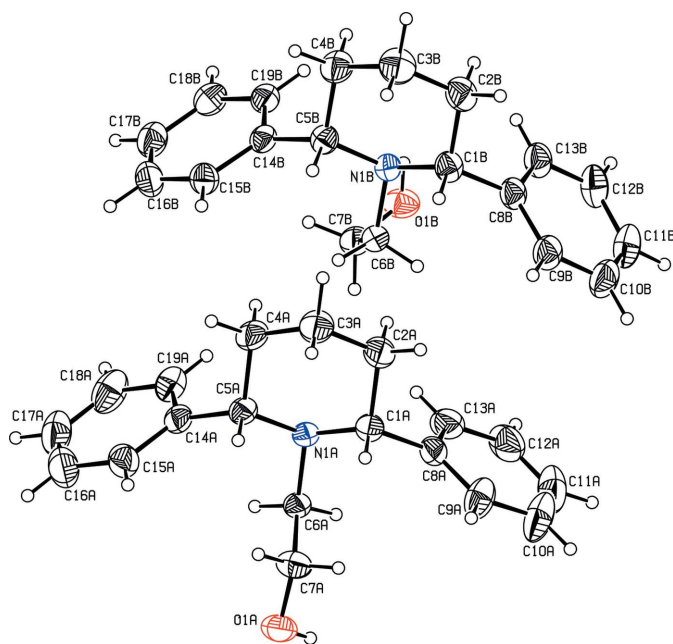


Figure 1
The asymmetric unit of (I), showing 30% probability displacement ellipsoids.

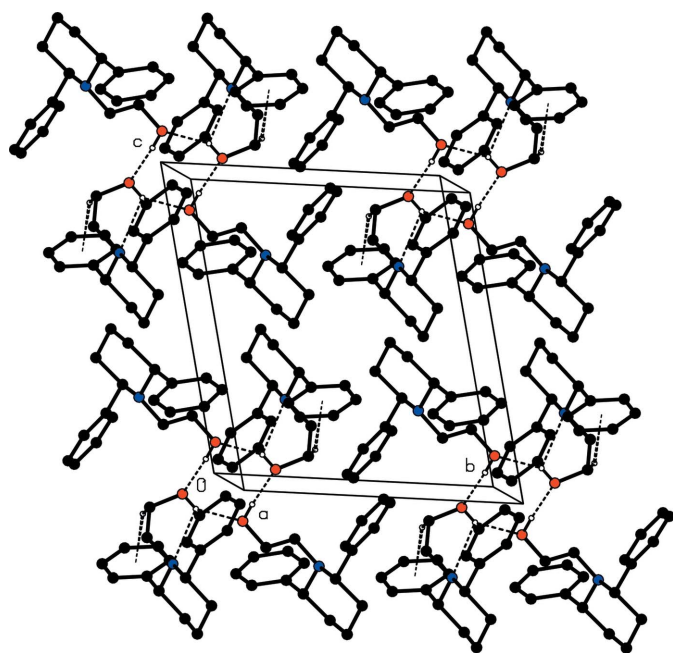


Figure 2
The crystal packing of (I), viewed down the *a* axis. H atoms which are not involved in hydrogen bonding have been omitted for clarity.

Intramolecular O—H...N and C—H... π interactions are observed only in molecule *B* and not in molecule *A*, because of the difference in the torsion angles N1A—C6A—C7A—O1A [174.1 (1)°] and N1B—C6B—C7B—O1B [50.7 (2)°] in molecules *A* and *B*, respectively. In the O—H...N interaction, atom O1B acts as donor to N1B, generating an *S*(5) ring motif. In the C—H... π interaction, atom C7B acts as a donor to the centroid, *C*_g, of the C14B—C19B ring, with an H7B2...*C*_g separation of 2.659 Å.

The crystal packing of (I) is stabilized by O—H...O intermolecular interactions (Table 2), generating *R*₄⁴(8) rings.

Experimental

To a homogenous solution of 1,3-diphenylpropane (0.992 mmol, 250 mg, 1 equivalent), ethanolamine (9.92 mmol, 605 mg, 10 equivalents) and polyethyleneglycol-200 (10 ml) in a 25 ml Erlenmeyer flask, 85% formic acid (9.92 mmol, 465 mg, 1 ml, 10 equivalents) was added at 273–278 K. The reaction mixture was then irradiated in a domestic microwave oven for 3 min at 370 W, after which time no 1,3-diphenylpropane was detected by 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 anhydrous Na₂SO₄ 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 (2–10%). After purification, compound (I) was obtained as a white crystalline solid (yield 76%, 212 mg). Single crystals were obtained by recrystallization from about 5% DCM in hexanes (b.p. 333–353 K).

Crystal data

C₁₉H₂₃NO
*M*_r = 281.38
 Triclinic, *P* $\bar{1}$
a = 11.7863 (9) Å
b = 12.2482 (9) Å
c = 12.8201 (10) Å
 α = 95.077 (1)°
 β = 107.637 (1)°
 γ = 109.768 (1)°

V = 1621.7 (2) Å³
Z = 4
*D*_x = 1.152 Mg m⁻³
 Mo *K* α radiation
 μ = 0.07 mm⁻¹
T = 293 (2) K
 Block, colourless
 0.26 × 0.24 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
 Absorption correction: none
 18837 measured reflections

7414 independent reflections
 5335 reflections with *I* > 2 σ (*I*)
*R*_{int} = 0.018
 θ _{max} = 28.0°

Refinement

Refinement on *F*²
R [*F*² > 2 σ (*F*²)] = 0.057
 wR (*F*²) = 0.173
S = 1.03
 7414 reflections
 381 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0901P)^2 + 0.2212P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.50 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

C1A—N1A	1.472 (2)	C1B—N1B	1.487 (2)
C5A—N1A	1.480 (2)	C5B—N1B	1.482 (2)
C6A—N1A	1.471 (2)	C6B—N1B	1.476 (2)
C7A—O1A	1.390 (2)	C7B—O1B	1.395 (2)
C6A—N1A—C1A	111.9 (1)	C6B—N1B—C5B	110.8 (1)
C6A—N1A—C5A	112.0 (1)	C6B—N1B—C1B	110.5 (1)
C1A—N1A—C5A	112.0 (1)	C5B—N1B—C1B	111.8 (1)
N1A—C6A—C7A—O1A	174.1 (1)	N1B—C6B—C7B—O1B	50.7 (2)
C7A—C6A—N1A—C1A	58.5 (2)	C7B—C6B—N1B—C1B	−139.7 (1)

Table 2

Hydrogen-bond geometry (Å, °).

C_g is the centroid of the C14B–C19B ring.

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O1B–H1B1···N1B	0.82	2.54	2.884 (2)	107
C7B–H7B2··· <i>C_g</i>	0.97	2.66	3.528 (2)	149
O1A–H1A1···O1B ⁱ	0.82	1.93	2.744 (2)	170
O1B–H1B1···O1A ⁱⁱ	0.82	2.09	2.816 (2)	148

Symmetry codes: (i) $-x + 1, -y + 1, -z$; (ii) $x, y - 1, z$.

All H atoms were positioned geometrically and allowed to ride on their parent C and O atoms, with the O–H distance fixed at 0.82 Å, with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$, and with C–H distances fixed in the range 0.93–0.98 Å, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{parent C})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; 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).

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