Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Ethyl-2,6-diphenylpiperidin-4-ol

A. Thiruvalluvar,[a]* S. Balamurugan,[a] J. Jayabharathi,[b] A. Manimekalai[b] and G. Rajarajan[b]

[a]Department of Physics, Rajah Serfoji Government College (Autonomous), Thanjavur 613 005, Tamilnadu, India, and [b]Department of Chemistry, Annamalai University, Annamalai, Nagar 608 002, Tamilnadu, India
Correspondence e-mail: athiru@vsnl.net

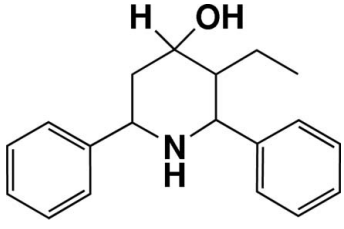
Received 24 April 2007; accepted 4 May 2007

Key indicators: single-crystal X-ray study; T = 160 K; mean σ(C–C) = 0.003 Å; R factor = 0.075; wR factor = 0.193; data-to-parameter ratio = 22.1.

In the title molecule, $C_{19}H_{23}NO$, the piperidine ring adopts a chair conformation. The two phenyl rings, and the hydroxy and ethyl groups attached to the piperidine ring, have equatorial orientations. The dihedral angle between the two phenyl rings is 54.5 (1)°. The hydroxy and amino H atoms are each disordered over two positions, with approximately equal site occupancies. Intermolecular O—H···O, O—H···N and N—H···O hydrogen bonds contribute to the stability of the crystal packing.

Related literature

The conformation of the title molecule was established by ^{1}H and ^{13}C NMR spectroscopy by Manimekalai & Rajarajan (1996). Pandiarajan *et al.* (2000) and Balamurugan *et al.* (2006, 2007) have reported the crystal structures of di-2-furylpiperidin-4-one derivatives, where the piperidine ring adopts chair and twist-boat conformations, respectively.



Experimental

Crystal data

$C_{19}H_{23}NO$
M_r = 281.38
Monoclinic, $P2_1/c$
a = 11.6611 (3) Å
b = 12.4948 (4) Å
c = 12.0089 (3) Å
β = 117.308 (1)°
V = 1554.73 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 160 (1) K
0.25 × 0.23 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
42996 measured reflections
4522 independent reflections
3412 reflections with $I > 2\sigma(I)$
R_{int} = 0.090

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.193
S = 1.13
4522 reflections
205 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···O4^{i}	0.86 (4)	1.98 (4)	2.841 (2)	176 (4)
O4—H4B···N1^{ii}	0.87 (6)	2.08 (6)	2.952 (3)	179 (4)
N1—H1A···O4^{iii}	0.89 (5)	2.09 (5)	2.952 (3)	179 (4)

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

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

AT thanks Dr A. Linden (Institute of Organic Chemistry at the University of Zurich) for his help with the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2239).

References

Balamurugan, S., Thiruvalluvar, A., Manimekalai, A., Selvaraju, K. & Maruthavanan, T. (2006). *Acta Cryst.* E**62**, o2005–o2006.
Balamurugan, S., Thiruvalluvar, A., Manimekalai, A., Selvaraju, K. & Maruthavanan, T. (2007). *Acta Cryst.* E**63**, o789–o791.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Manimekalai, A. & Rajarajan, G. (1996). *Indian J. Chem. Sect. B*, **35**, 517–524.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. London: Academic Press.
Pandiarajan, K., Manimekalai, A. & Rajarajan, G. (2000). *Indian J. Chem. Sect. B*, **39**, 923–928.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

  doi:10.1107/S1600536807022131

supplementary materials

Acta Cryst. (2007). E**63**, o2886 [**doi:10.1107/S1600536807022131**]

3-Ethyl-2,6-diphenylpiperidin-4-ol

A. Thiruvalluvar, S. Balamurugan, J. Jayabharathi, A. Manimekalai and G. Rajarajan

Comment

The conformation of (I) was established by ^{1}H and ^{13}C NMR sepectroscopy by Manimekalai & Rajarajan, 1996; Pandiarajan, *et al.*, 2000. Balamurugan *et al.*, 2006, 2007 have reported crystal structures of di-2-furylpiperidin-4-one derivatives, wherein the piperidine ring adopts a chair and a twist-boat conformations respectively. In the title compound,(I), (Fig. 1), piperidine ring adopts a chair coformation. The hydroxy group in the 4 position, the ethyl group at position 3 and the phenyl rings at positions 2 and 6 have equatorial orientations. The dihedral angle between the two phenyl rings is 54.5 (1)°. The hydroxy and amino H atoms are disordered between two positions each, providing an existence of intermolecular O–H···O, O–H···N and N–H···O hydrogen bonds, which contribute to the stability of crystal packing.

Experimental

The title compound was prepared from t(3)-ethyl-r(2),c(6)-diphenylpiperidin-4-one by sodium/alcohol reduction, followed by chromatographic separation over neutral alumina, and its conformation was established by ^{1}H and ^{13}C NMR sepectroscopy (Manimekalai & Rajarajan, 1996; Pandiarajan *et al.*, 2000). The compound was recrystallized from petroleum-ether (333-353 K).

Refinement

The C-bound H atoms were positioned geometrically and allowed to ride on their parent atoms with C—H = 0.95-1.00 Å and U_{iso}(H) = 1.2-1.5U_{eq} (parent atom). The H atoms attached to N1 and O4 were treated as disordered between two positions each, with the positions located in a difference map, and refined with bond restraints O—H = 0.85 (4) Å, N—H = 0.86 (4) Å, and with Uiso(H) = 1.5Ueq(N, O). The refined occupancies were 0.48 (4) and 0.52 (4) for atoms H1A and H1B [N1], respectively, and 0.57 (4) and 0.43 (4) for atoms H4A and H4B [O4], respectively.

Figures

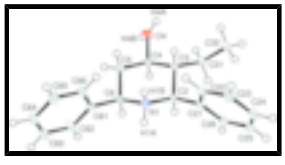


Fig. 1. View of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms attached to N1 and O4 are disordered.

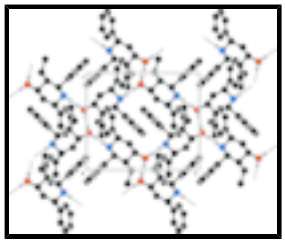


Fig. 2. The molecular packing of (I), viewed down the a axis, showing the hydrogen bonds (dashed lines).

3-Ethyl-2,6-diphenylpiperidin-4-ol

Crystal data

$C_{19}H_{23}NO$	$F_{000} = 608$
$M_r = 281.38$	$D_x = 1.202$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 394(1) K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 11.6611$ (3) Å	Cell parameters from 4693 reflections
$b = 12.4948$ (4) Å	$\theta = 2.0$–30.0º
$c = 12.0089$ (3) Å	$\mu = 0.07$ mm^{-1}
$\beta = 117.308$ (1)º	$T = 160$ (1) K
$V = 1554.73$ (7) Å^3	Needles, colourless
$Z = 4$	0.25 × 0.23 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer	4522 independent reflections
Radiation source: Nonius FR590 sealed tube generator	3412 reflections with $I > 2\sigma(I)$
Monochromator: horizontally mounted graphite crystal	$R_{int} = 0.090$
Detector resolution: 9 pixels mm^{-1}	$\theta_{max} = 30.0$º
$T = 160(1)$ K	$\theta_{min} = 2.5$º
φ and ω scans with κ offsets	$h = -16 \rightarrow 16$
Absorption correction: none	$k = -17 \rightarrow 17$
42996 measured reflections	$l = -16 \rightarrow 15$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.075$	$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 1.6386P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.193$	$(\Delta/\sigma)_{max} = <0.001$
$S = 1.13$	$\Delta\rho_{max} = 0.45$ e Å^{-3}
4522 reflections	$\Delta\rho_{min} = -0.27$ e Å^{-3}
205 parameters	Extinction correction: none
4 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Special details

Experimental. Solvent used: petroleumether/benzene mixture Cooling Device: Oxford Cryosystems Cryostream 700 Crystal mount: glued on a glass fibre Mosaicity (deg.): 0.532 (2) Frames collected: 362 Seconds exposure per frame: 80 Degrees rotation per frame: 2.0 Crystal-Detector distance (mm): 30.0

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O4	0.50486 (15)	0.52095 (11)	0.11834 (14)	0.0221 (4)	
N1	0.51515 (17)	0.20190 (14)	0.23352 (16)	0.0201 (5)	
C2	0.38754 (18)	0.24572 (15)	0.14651 (17)	0.0160 (5)	
C3	0.39891 (18)	0.34789 (15)	0.07940 (17)	0.0150 (5)	
C4	0.48487 (19)	0.42896 (15)	0.17787 (17)	0.0175 (5)	
C5	0.61368 (19)	0.38079 (15)	0.26871 (18)	0.0177 (5)	
C6	0.59507 (18)	0.27918 (15)	0.33003 (17)	0.0164 (5)	
C21	0.30935 (18)	0.15937 (16)	0.05442 (17)	0.0168 (5)	
C22	0.3612 (2)	0.10140 (17)	−0.01094 (19)	0.0209 (6)	
C23	0.2893 (2)	0.02414 (19)	−0.0986 (2)	0.0271 (6)	
C24	0.1628 (2)	0.00416 (19)	−0.1225 (2)	0.0294 (7)	
C25	0.1097 (2)	0.0617 (2)	−0.0590 (2)	0.0288 (6)	
C26	0.18262 (19)	0.13811 (18)	0.02957 (19)	0.0225 (6)	
C31	0.26640 (19)	0.39729 (18)	−0.00634 (19)	0.0227 (6)	
C32	0.2082 (2)	0.3631 (2)	−0.1436 (2)	0.0301 (7)	
C61	0.72247 (19)	0.22879 (15)	0.41831 (17)	0.0172 (5)	
C62	0.7642 (2)	0.22886 (18)	0.54673 (19)	0.0252 (6)	
C63	0.8835 (2)	0.1862 (2)	0.6285 (2)	0.0310 (7)	
C64	0.9625 (2)	0.14282 (19)	0.5829 (2)	0.0287 (6)	
C65	0.9210 (2)	0.1400 (2)	0.4550 (2)	0.0284 (7)	
C66	0.8020 (2)	0.18294 (18)	0.37324 (19)	0.0239 (6)	
H1B	0.555 (4)	0.178 (4)	0.194 (4)	0.0302*	0.52 (4)
H2	0.34194	0.26543	0.19666	0.0192*	
H3	0.44155	0.32815	0.02657	0.0180*	
H4	0.43928	0.45239	0.22678	0.0209*	
H4A	0.499 (5)	0.511 (4)	0.045 (3)	0.0331*	0.57 (4)
H5A	0.66340	0.43388	0.33451	0.0211*	
H5B	0.66391	0.36334	0.22335	0.0211*	
H6	0.54875	0.29914	0.37943	0.0196*	
H22	0.44749	0.11495	0.00469	0.0251*	

H23	0.32642	−0.01483	−0.14189	0.0326*	
H24	0.11311	−0.04866	−0.18203	0.0353*	
H25	0.02292	0.04897	−0.07596	0.0345*	
H26	0.14553	0.17620	0.07356	0.0271*	
H31A	0.20547	0.37772	0.02665	0.0273*	
H31B	0.27507	0.47620	−0.00217	0.0273*	
H32A	0.26873	0.37997	−0.17687	0.0451*	
H32B	0.12698	0.40145	−0.19204	0.0451*	
H32C	0.19170	0.28586	−0.14992	0.0451*	
H62	0.71058	0.25846	0.57910	0.0303*	
H63	0.91086	0.18686	0.71622	0.0371*	
H64	1.04482	0.11510	0.63896	0.0344*	
H65	0.97405	0.10851	0.42304	0.0342*	
H66	0.77455	0.18108	0.28555	0.0287*	
H1A	0.508 (5)	0.149 (3)	0.277 (4)	0.0302*	0.48 (4)
H4B	0.500 (6)	0.575 (3)	0.162 (5)	0.0331*	0.43 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O4	0.0360 (9)	0.0117 (7)	0.0198 (7)	−0.0005 (6)	0.0139 (7)	0.0003 (5)
N1	0.0201 (8)	0.0135 (8)	0.0193 (8)	0.0015 (7)	0.0026 (7)	−0.0004 (6)
C2	0.0159 (9)	0.0172 (9)	0.0148 (8)	0.0005 (7)	0.0070 (7)	0.0007 (7)
C3	0.0167 (9)	0.0154 (9)	0.0140 (8)	0.0017 (7)	0.0079 (7)	0.0015 (7)
C4	0.0257 (10)	0.0119 (9)	0.0157 (9)	0.0020 (7)	0.0103 (8)	0.0014 (7)
C5	0.0204 (9)	0.0143 (9)	0.0159 (9)	−0.0020 (7)	0.0063 (8)	−0.0008 (7)
C6	0.0193 (9)	0.0159 (9)	0.0138 (8)	−0.0008 (7)	0.0075 (7)	−0.0007 (7)
C21	0.0173 (9)	0.0164 (9)	0.0156 (9)	−0.0006 (7)	0.0065 (7)	0.0040 (7)
C22	0.0197 (9)	0.0211 (10)	0.0215 (10)	−0.0015 (8)	0.0090 (8)	−0.0005 (8)
C23	0.0286 (11)	0.0278 (12)	0.0225 (10)	−0.0029 (9)	0.0096 (9)	−0.0044 (9)
C24	0.0306 (12)	0.0267 (12)	0.0214 (10)	−0.0096 (9)	0.0038 (9)	−0.0013 (9)
C25	0.0178 (10)	0.0369 (13)	0.0245 (10)	−0.0074 (9)	0.0036 (8)	0.0066 (9)
C26	0.0176 (9)	0.0273 (11)	0.0212 (10)	0.0008 (8)	0.0076 (8)	0.0048 (8)
C31	0.0184 (9)	0.0238 (11)	0.0219 (10)	0.0040 (8)	0.0057 (8)	0.0054 (8)
C32	0.0223 (11)	0.0370 (13)	0.0211 (10)	0.0014 (9)	0.0015 (9)	0.0065 (9)
C61	0.0196 (9)	0.0144 (9)	0.0152 (9)	−0.0032 (7)	0.0059 (7)	0.0000 (7)
C62	0.0290 (11)	0.0275 (11)	0.0156 (9)	0.0040 (9)	0.0072 (8)	−0.0001 (8)
C63	0.0313 (12)	0.0383 (13)	0.0138 (9)	0.0041 (10)	0.0022 (9)	0.0010 (9)
C64	0.0214 (10)	0.0321 (12)	0.0223 (10)	0.0032 (9)	0.0013 (9)	0.0053 (9)
C65	0.0214 (10)	0.0371 (13)	0.0264 (11)	0.0043 (9)	0.0106 (9)	0.0025 (9)
C66	0.0233 (10)	0.0326 (12)	0.0162 (9)	0.0023 (9)	0.0094 (8)	0.0023 (8)

Geometric parameters (Å, °)

O4—C4	1.428 (2)	C63—C64	1.380 (4)
O4—H4A	0.86 (4)	C64—C65	1.383 (3)
O4—H4B	0.87 (5)	C65—C66	1.388 (3)
N1—C6	1.467 (3)	C2—H2	1.0000
N1—C2	1.476 (3)	C3—H3	1.0000

N1—H1A	0.87 (4)	C4—H4	1.0000
N1—H1B	0.86 (5)	C5—H5A	0.9900
C2—C3	1.548 (3)	C5—H5B	0.9900
C2—C21	1.514 (3)	C6—H6	1.0000
C3—C31	1.539 (3)	C22—H22	0.9500
C3—C4	1.530 (3)	C23—H23	0.9500
C4—C5	1.518 (3)	C24—H24	0.9500
C5—C6	1.532 (3)	C25—H25	0.9500
C6—C61	1.511 (3)	C26—H26	0.9500
C21—C26	1.392 (3)	C31—H31A	0.9900
C21—C22	1.394 (3)	C31—H31B	0.9900
C22—C23	1.390 (3)	C32—H32A	0.9800
C23—C24	1.390 (4)	C32—H32B	0.9800
C24—C25	1.384 (3)	C32—H32C	0.9800
C25—C26	1.391 (3)	C62—H62	0.9500
C31—C32	1.528 (3)	C63—H63	0.9500
C61—C62	1.388 (3)	C64—H64	0.9500
C61—C66	1.394 (3)	C65—H65	0.9500
C62—C63	1.389 (3)	C66—H66	0.9500
O4···O4^{i}	2.841 (2)	H4···H23^{vi}	2.5900
O4···N1^{ii}	2.952 (2)	H4···H31B	2.5500
O4···H1A^{ii}	2.08 (4)	H4···H1A^{ii}	2.5400
O4···H31B	2.4600	H4···H2	2.5500
O4···H3^{i}	2.8200	H4A···O4^{i}	1.98 (4)
O4···H4A^{i}	1.98 (4)	H4A···C3^{i}	2.90 (5)
O4···H32A^{i}	2.7000	H4A···C4^{i}	2.87 (4)
N1···O4^{iii}	2.952 (2)	H4A···H3^{i}	2.4100
N1···H22	2.7100	H4A···C31	2.87 (6)
N1···H66	2.8100	H4A···H3	2.3600
N1···H4B^{iii}	2.08 (5)	H4A···H31B	2.4400
C21···C32	3.309 (3)	H4A···H4A^{i}	1.13 (6)
C24···C62^{iii}	3.560 (3)	H4B···N1^{ii}	2.08 (5)
C26···C32	3.590 (3)	H4B···C2^{ii}	2.97 (5)
C26···C31	3.466 (3)	H4B···C61^{ii}	3.01 (6)
C31···C26	3.466 (3)	H4B···H1B^{ii}	2.47 (8)
C32···C21	3.309 (3)	H4B···C6^{ii}	2.80 (5)
C32···C26	3.590 (3)	H5A···C21^{ii}	3.0700
C62···C24^{ii}	3.560 (3)	H5A···C22^{ii}	3.0800
C2···H4B^{iii}	2.97 (5)	H5B···C66	2.8700
C3···H4A^{i}	2.90 (5)	H5B···H1B	2.5900
C4···H1A^{ii}	2.80 (4)	H5B···H66	2.5500
C4···H4A^{i}	2.87 (4)	H5B···C62^{viii}	3.0800
C5···H66	3.0700	H5B···H62^{viii}	2.5500
C6···H4B^{iii}	2.80 (5)	H6···H4	2.5500

C21···H5A^{iii}	3.0700	H6···H62	2.3300
C21···H32C	2.7000	H6···H22^{vi}	2.5400
C21···H31A	2.9400	H6···H2	2.4400
C22···H32C	3.0000	H22···H1B	2.1800
C22···H3	2.9500	H22···H6^{viii}	2.5400
C22···H5A^{iii}	3.0800	H22···N1	2.7100
C22···H1B	2.64 (5)	H23···H4^{viii}	2.5900
C24···H64^{iv}	2.9000	H24···H32B^{ix}	2.5700
C25···H25^{v}	3.0400	H25···C25^{v}	3.0400
C25···H63^{iv}	3.0600	H25···H25^{v}	2.4500
C26···H32C	2.8800	H26···H2	2.3600
C26···H31A	3.0100	H31A···C65^{x}	3.0300
C31···H4A	2.87 (6)	H31A···C21	2.9400
C61···H4B^{iii}	3.01 (6)	H31A···C26	3.0100
C62···H5B^{vi}	3.0800	H31A···H65^{x}	2.4000
C62···H66^{vi}	3.0300	H31A···H2	2.3800
C65···H31A^{vii}	3.0300	H31B···H4	2.5500
C66···H1B	2.69 (5)	H31B···H4A	2.4400
C66···H5B	2.8700	H31B···O4	2.4600
H1A···O4^{iii}	2.08 (4)	H32A···O4^{i}	2.7000
H1A···C4^{iii}	2.80 (4)	H32A···H3	2.4400
H1A···H4^{iii}	2.5400	H32B···H24^{xi}	2.5700
H1B···C22	2.64 (5)	H32C···C21	2.7000
H1B···C66	2.69 (5)	H32C···C22	3.0000
H1B···H5B	2.5900	H32C···C26	2.8800
H1B···H4B^{iii}	2.47 (8)	H62···H6	2.3300
H1B···H22	2.1800	H62···H5B^{vi}	2.5500
H1B···H66	2.2800	H62···H66^{vi}	2.3600
H2···H4	2.5500	H63···C25^{xii}	3.0600
H2···H6	2.4400	H64···C24^{xii}	2.9000
H2···H26	2.3600	H65···H31A^{vii}	2.4000
H2···H31A	2.3800	H66···C5	3.0700
H3···O4^{i}	2.8200	H66···H1B	2.2800
H3···H4A^{i}	2.4100	H66···N1	2.8100
H3···H32A	2.4400	H66···C62^{viii}	3.0300
H3···C22	2.9500	H66···H62^{viii}	2.3600
H3···H4A	2.3600	H66···H5B	2.5500
H4···H6	2.5500		
C4—O4—H4A	116 (3)	O4—C4—H4	108.00
C4—O4—H4B	105 (4)	C3—C4—H4	108.00
C2—N1—C6	112.56 (16)	C5—C4—H4	108.00
C2—N1—H1A	111 (4)	C4—C5—H5A	109.00
C6—N1—H1B	111 (3)	C4—C5—H5B	109.00

C6—N1—H1A	103 (3)	C6—C5—H5A	109.00
C2—N1—H1B	111 (3)	C6—C5—H5B	109.00
N1—C2—C3	112.04 (18)	H5A—C5—H5B	108.00
N1—C2—C21	109.05 (16)	N1—C6—H6	108.00
C3—C2—C21	111.97 (15)	C5—C6—H6	108.00
C2—C3—C31	112.31 (18)	C61—C6—H6	108.00
C4—C3—C31	110.62 (16)	C21—C22—H22	119.00
C2—C3—C4	109.02 (15)	C23—C22—H22	119.00
O4—C4—C5	110.11 (18)	C22—C23—H23	120.00
O4—C4—C3	110.21 (15)	C24—C23—H23	120.00
C3—C4—C5	111.98 (16)	C23—C24—H24	120.00
C4—C5—C6	111.24 (18)	C25—C24—H24	120.00
N1—C6—C5	110.14 (15)	C24—C25—H25	120.00
N1—C6—C61	110.11 (16)	C26—C25—H25	120.00
C5—C6—C61	111.84 (18)	C21—C26—H26	120.00
C2—C21—C26	121.11 (19)	C25—C26—H26	120.00
C2—C21—C22	120.7 (2)	C3—C31—H31A	109.00
C22—C21—C26	118.18 (19)	C3—C31—H31B	109.00
C21—C22—C23	121.4 (2)	C32—C31—H31A	109.00
C22—C23—C24	119.7 (2)	C32—C31—H31B	109.00
C23—C24—C25	119.6 (2)	H31A—C31—H31B	108.00
C24—C25—C26	120.4 (2)	C31—C32—H32A	109.00
C21—C26—C25	120.7 (2)	C31—C32—H32B	110.00
C3—C31—C32	114.99 (19)	C31—C32—H32C	109.00
C6—C61—C62	120.7 (2)	H32A—C32—H32B	109.00
C6—C61—C66	120.95 (17)	H32A—C32—H32C	109.00
C62—C61—C66	118.4 (2)	H32B—C32—H32C	109.00
C61—C62—C63	120.8 (2)	C61—C62—H62	120.00
C62—C63—C64	120.3 (2)	C63—C62—H62	120.00
C63—C64—C65	119.6 (2)	C62—C63—H63	120.00
C64—C65—C66	120.2 (2)	C64—C63—H63	120.00
C61—C66—C65	120.77 (19)	C63—C64—H64	120.00
N1—C2—H2	108.00	C65—C64—H64	120.00
C3—C2—H2	108.00	C64—C65—H65	120.00
C21—C2—H2	108.00	C66—C65—H65	120.00
C2—C3—H3	108.00	C61—C66—H66	120.00
C4—C3—H3	108.00	C65—C66—H66	120.00
C31—C3—H3	108.00		
C6—N1—C2—C3	58.2 (2)	C4—C5—C6—N1	55.8 (2)
C6—N1—C2—C21	−177.24 (17)	N1—C6—C61—C62	−126.0 (2)
C2—N1—C6—C5	−57.9 (2)	N1—C6—C61—C66	55.2 (3)
C2—N1—C6—C61	178.29 (17)	C5—C6—C61—C62	111.2 (2)
C21—C2—C3—C4	−176.98 (18)	C5—C6—C61—C66	−67.6 (2)
N1—C2—C3—C4	−54.1 (2)	C2—C21—C22—C23	−178.01 (19)
N1—C2—C3—C31	−177.06 (16)	C26—C21—C22—C23	−0.1 (3)
N1—C2—C21—C26	131.7 (2)	C2—C21—C26—C25	177.32 (19)
C3—C2—C21—C22	74.1 (2)	C22—C21—C26—C25	−0.6 (3)
C3—C2—C21—C26	−103.7 (2)	C21—C22—C23—C24	0.3 (3)
C21—C2—C3—C31	60.1 (2)	C22—C23—C24—C25	0.2 (3)

N1—C2—C21—C22	−50.5 (2)	C23—C24—C25—C26	−0.9 (3)
C31—C3—C4—O4	−60.3 (2)	C24—C25—C26—C21	1.1 (3)
C2—C3—C4—O4	175.72 (18)	C6—C61—C62—C63	−177.6 (2)
C2—C3—C4—C5	52.8 (2)	C66—C61—C62—C63	1.3 (3)
C4—C3—C31—C32	143.22 (19)	C6—C61—C66—C65	177.8 (2)
C31—C3—C4—C5	176.75 (18)	C62—C61—C66—C65	−1.1 (3)
C2—C3—C31—C32	−94.7 (2)	C61—C62—C63—C64	−0.1 (4)
O4—C4—C5—C6	−177.68 (15)	C62—C63—C64—C65	−1.4 (4)
C3—C4—C5—C6	−54.7 (2)	C63—C64—C65—C66	1.6 (4)
C4—C5—C6—C61	178.59 (16)	C64—C65—C66—C61	−0.4 (4)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) −*x*+1, *y*+1/2, −*z*+1/2; (iii) −*x*+1, *y*−1/2, −*z*+1/2; (iv) *x*−1, *y*, *z*−1; (v) −*x*, −*y*, −*z*; (vi) *x*, −*y*+1/2, *z*+1/2; (vii) *x*+1, −*y*+1/2, *z*+1/2; (viii) *x*, −*y*+1/2, *z*−1/2; (ix) −*x*, *y*−1/2, −*z*−1/2; (x) *x*−1, −*y*+1/2, *z*−1/2; (xi) −*x*, *y*+1/2, −*z*−1/2; (xii) *x*+1, *y*, *z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4A···O4^{i}	0.86 (4)	1.98 (4)	2.841 (2)	176 (4)
O4—H4B···N1^{ii}	0.87 (6)	2.08 (6)	2.952 (3)	179 (4)
N1—H1A···O4^{iii}	0.89 (5)	2.09 (5)	2.952 (3)	179 (4)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) −*x*+1, *y*+1/2, −*z*+1/2; (iii) −*x*+1, *y*−1/2, −*z*+1/2.

Fig. 1

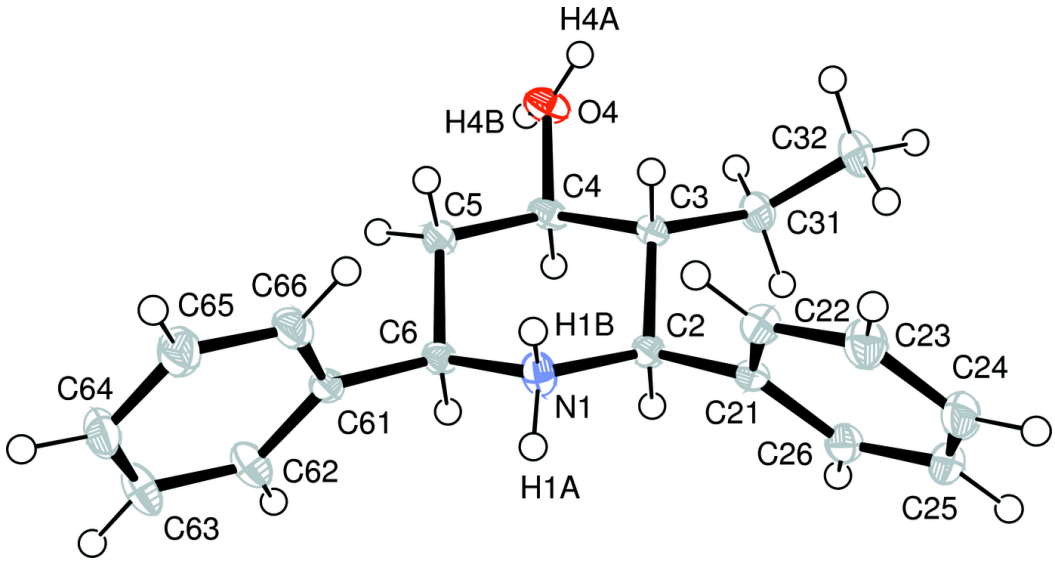

H4A
H4B
O4
C32
C5
C4
C3
C31
C66
C65
C6
H1B
C2
C22
C23
C24
C64
C61
N1
C21
C26
C62
H1A
C25
C63

Fig. 2