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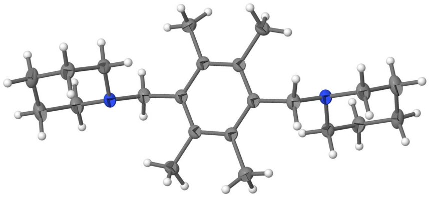
1,1′-[(2,3,5,6-Tetramethyl-1,4-phenylene)bis(methylene)]dipiperidine

P. Selvarathy Grace,[a] Jeyaraman Selvaraj Nirmalram,[b] B. Ravindran Durai Nayagam[a]* and Dieter Schollmeyer[c]

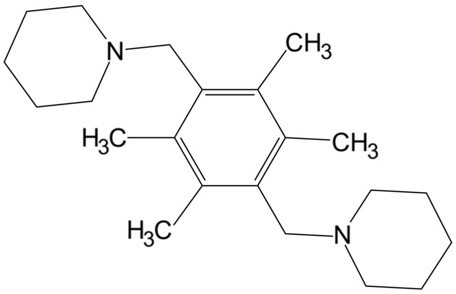
[a]Department of Chemistry, Popes College affiliated to Manonmaniam Sundaranar University, Tirunelveli, Tamil Nadu 627 012, India, [b]Department of Chemistry, Ponnaiyah Ramajayam Institute of Science and Technology, Vallam, Thanjavur 613 403, TamilNadu, India, and [c]University of Mainz, Institut of Organic Chemistry, Duesbergweg 10-14, 55128 Mainz, Germany. *Correspondence e-mail: bravidurai@gmail.com

The asymmetric unit of the title compound, $C_{22}H_{36}N_2$, comprises one half-molecule, the other half being generated by a center of inversion. The piperidine ring adopts a chair conformation, with the exocyclic N—C bond in an equatorial orientation. A short intramolecular C—H···N hydrogen bond occurs and forms an $S(6)$ motif. No directional interactions beyond van der Waals contacts are observed between the molecules, which form a wave-like supramolecular architecture.

3D view



Chemical scheme



Structure description

Some piperidine derivatives possess significant biological and medicinal properties including insulin normalization, addiction therapeutics (Kozikowski *et al.*, 1998) and local anaesthesia (McElvain & Carney, 1946). Here we report the synthesis and crystal structure of a new piperidine-substituted durene.

The asymmetric unit is made up of one half-molecule, the other half being generated by inversion (symmetry code: $1 - x, 1 - y, 1 - z$; see Fig. 1). The piperidine rings adopt a chair conformation with puckering parameters Q = 0.5765 (16) Å, Θ =178.20 (15)° φ = 181 (5)°. The dihedral angle between the phenyl (C8/C9/C11/C8a/C9a/C11*a*) and piperidine (N1/C2–C6) rings is 73.66 (7)°. The weak intramolecular C12—H12*B*···N1 hydrogen bond (Table 1) forms an $S(6)$ motif. In the crystal, adjacent molecules are aggregated by weak van der Waals interactions, leading to a wave-like supramolecular architecture (Fig. 2) extending along *b*-axis direction.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C12—H12$B\cdots$N1	0.94 (2)	2.59 (2)	3.2204 (18)	124.8 (14)

Synthesis and crystallization

A mixture of piperidine hydrochloride (0.242 g, 2 mmol) in sodium ethoxide solution and 1,4-bis(bromomethyl)durene (0.320 g, 1 mmol) in ethanol and water (15 ml) were heated at 333 K with continuous stirring for about 4 h. The mixture was kept aside for slow evaporation. After two weeks, colourless block-shaped crystals (m.p. 462 K) suitable for single-crystal X-ray diffraction were formed.

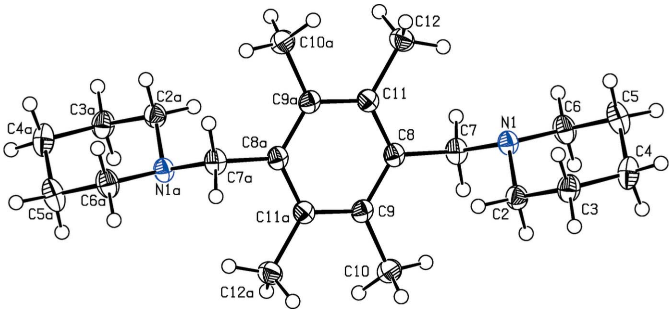


Figure 1
The molecular structure of the title compound with the atom labelling and displacement ellipsoids drawn at the 50% probability level [symmetry code: (a) $1 - x, 1 - y, 1 - z$].

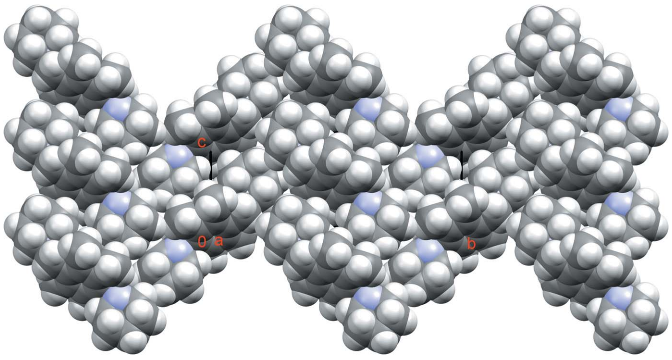


Figure 2
Part of the crystal packing showing the wave-like architecture. View is along the a axis.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{36}N_2$
M_r	328.53
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	173
a, b, c (Å)	5.6271 (4), 21.1764 (14), 8.2787 (5)
β (°)	105.860 (2)
V (Å^3)	948.95 (11)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.07
Crystal size (mm)	0.45 × 0.17 × 0.15
Data collection	
Diffractometer	Bruker SMART APEXII
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6578, 2237, 1779
$R_{\rm int}$	0.027
$(\sin \theta/\lambda)_{\rm max}$ (Å^{-1})	0.655
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.118, 1.03
No. of reflections	2237
No. of parameters	172
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{\rm max}$, $\Delta\rho_{\rm min}$ (e Å^{-3})	0.28, −0.16

Computer programs: *APEX2* and *SAINT* (Bruker, 2004), *SIR2004* (Burla *et al.*, 2005), *SHELXL2018* (Sheldrick, 2015), *ORTEPII* (Johnson, 1976), *Mercury* (Macrae *et al.* (2008) and *POV-RAY for Windows* (Cason, 2004).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2018). **3**, x181237 [https://doi.org/10.1107/S2414314618012373]

1,1′-[(2,3,5,6-Tetramethyl-1,4-phenylene)bis(methylene)]dipiperidine

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Crystal data

$C_{22}H_{36}N_2$
$M_r = 328.53$
Monoclinic, $P2_1/n$
$a = 5.6271$ (4) Å
$b = 21.1764$ (14) Å
$c = 8.2787$ (5) Å
$\beta = 105.860$ (2)°
$V = 948.95$ (11) Å^3
$Z = 2$
$F(000) = 364$
$D_x = 1.150$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1751 reflections
$\theta = 2.7–27.7°$
$\mu = 0.07$ mm^{-1}
$T = 173$ K
Block, colourless
0.45 × 0.17 × 0.15 mm

Data collection

Bruker SMART APEXII diffractometer
Radiation source: sealed Tube
Graphite monochromator
CCD scan
6578 measured reflections
2237 independent reflections
1779 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\rm max} = 27.7°$, $\theta_{\rm min} = 1.9°$
$h = -7\rightarrow6$
$k = -27\rightarrow16$
$l = -9\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.118$
$S = 1.03$
2237 reflections
172 parameters
0 restraints
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.2324P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.28$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. The H atoms were added at positions obtained from difference Fourier maps and refined isotropically.

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

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
N1	0.48878 (19)	0.63789 (5)	0.20943 (13)	0.0239 (3)
C2	0.4075 (2)	0.59637 (6)	0.06304 (16)	0.0268 (3)
H2A	0.221 (3)	0.6020 (6)	0.0122 (18)	0.029 (3)*
H2B	0.434 (3)	0.5516 (7)	0.1021 (18)	0.029 (3)*
C3	0.5439 (3)	0.61062 (6)	−0.06807 (17)	0.0305 (3)
H3A	0.487 (3)	0.5819 (8)	−0.166 (2)	0.039 (3)*
H3B	0.725 (3)	0.6027 (7)	−0.018 (2)	0.039 (3)*
C4	0.5071 (3)	0.67927 (7)	−0.12320 (19)	0.0363 (3)
H4A	0.331 (3)	0.6873 (8)	−0.179 (2)	0.048 (3)*
H4B	0.605 (3)	0.6902 (8)	−0.201 (2)	0.048 (3)*
C5	0.5808 (3)	0.72219 (7)	0.0303 (2)	0.0380 (4)
H5A	0.765 (3)	0.7182 (8)	0.083 (2)	0.047 (3)*
H5B	0.549 (3)	0.7677 (9)	−0.003 (2)	0.047 (3)*
C6	0.4436 (3)	0.70403 (6)	0.15832 (18)	0.0320 (3)
H6A	0.497 (3)	0.7317 (7)	0.262 (2)	0.037 (3)*
H6B	0.258 (3)	0.7116 (7)	0.1108 (19)	0.037 (3)*
C7	0.3611 (2)	0.62262 (6)	0.33824 (16)	0.0267 (3)
H7A	0.178 (3)	0.6271 (6)	0.2908 (18)	0.028 (2)*
H7B	0.407 (3)	0.6563 (7)	0.4244 (18)	0.028 (2)*
C8	0.4317 (2)	0.55846 (6)	0.41941 (15)	0.0227 (3)
C9	0.2674 (2)	0.50691 (6)	0.38251 (14)	0.0232 (3)
C10	0.0179 (3)	0.51192 (7)	0.25473 (18)	0.0312 (3)
H10A	0.015 (4)	0.4889 (11)	0.152 (3)	0.088 (4)*
H10B	−0.024 (4)	0.5535 (12)	0.220 (3)	0.088 (4)*
H10C	−0.114 (5)	0.4943 (10)	0.296 (3)	0.088 (4)*
C11	0.6640 (2)	0.55124 (6)	0.53631 (15)	0.0231 (3)
C12	0.8452 (3)	0.60566 (7)	0.57599 (18)	0.0307 (3)
H12A	0.882 (3)	0.6199 (8)	0.695 (2)	0.056 (3)*
H12B	0.791 (4)	0.6411 (9)	0.508 (2)	0.056 (3)*
H12C	1.002 (4)	0.5941 (9)	0.561 (2)	0.056 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0289 (5)	0.0205 (5)	0.0239 (5)	0.0011 (4)	0.0102 (4)	0.0009 (4)
C2	0.0328 (7)	0.0242 (6)	0.0244 (6)	−0.0008 (5)	0.0095 (5)	0.0001 (5)
C3	0.0380 (7)	0.0309 (7)	0.0255 (7)	0.0034 (6)	0.0136 (6)	0.0014 (5)
C4	0.0427 (8)	0.0372 (8)	0.0339 (8)	0.0082 (6)	0.0187 (7)	0.0127 (6)
C5	0.0491 (9)	0.0247 (7)	0.0477 (9)	0.0014 (6)	0.0256 (7)	0.0074 (6)
C6	0.0409 (8)	0.0208 (6)	0.0375 (8)	0.0032 (5)	0.0164 (6)	0.0021 (5)
C7	0.0316 (7)	0.0248 (6)	0.0269 (7)	0.0036 (5)	0.0134 (5)	0.0009 (5)
C8	0.0275 (6)	0.0243 (6)	0.0194 (6)	0.0005 (5)	0.0117 (5)	−0.0014 (4)
C9	0.0255 (6)	0.0283 (6)	0.0180 (6)	0.0004 (5)	0.0098 (5)	−0.0016 (5)
C10	0.0286 (7)	0.0359 (7)	0.0274 (7)	−0.0018 (5)	0.0049 (5)	0.0015 (6)
C11	0.0282 (6)	0.0249 (6)	0.0194 (6)	−0.0024 (5)	0.0117 (5)	−0.0036 (4)

C12	0.0337 (7)	0.0290 (7)	0.0294 (7)	−0.0073 (5)	0.0085 (6)	−0.0030 (5)

Geometric parameters (Å, º)

N1—C6	1.4652 (16)	C6—H6B	1.024 (16)
N1—C2	1.4655 (16)	C7—C8	1.5198 (17)
N1—C7	1.4753 (16)	C7—H7A	1.003 (15)
C2—C3	1.5207 (18)	C7—H7B	0.991 (15)
C2—H2A	1.026 (15)	C8—C11	1.4060 (17)
C2—H2B	1.000 (14)	C8—C9	1.4090 (17)
C3—C4	1.5209 (19)	C9—C11^{i}	1.4055 (17)
C3—H3A	0.991 (17)	C9—C10	1.5131 (18)
C3—H3B	1.003 (17)	C10—H10A	0.98 (2)
C4—C5	1.525 (2)	C10—H10B	0.94 (2)
C4—H4A	0.987 (18)	C10—H10C	0.97 (2)
C4—H4B	0.984 (18)	C11—C12	1.5143 (17)
C5—C6	1.521 (2)	C12—H12A	0.996 (19)
C5—H5A	1.013 (18)	C12—H12B	0.94 (2)
C5—H5B	1.003 (18)	C12—H12C	0.957 (19)
C6—H6A	1.017 (16)		

C6—N1—C2	110.14 (10)	N1—C6—H6B	109.9 (9)
C6—N1—C7	109.74 (10)	C5—C6—H6B	110.6 (9)
C2—N1—C7	111.27 (10)	H6A—C6—H6B	105.6 (12)
N1—C2—C3	111.53 (11)	N1—C7—C8	113.30 (10)
N1—C2—H2A	108.8 (8)	N1—C7—H7A	110.4 (8)
C3—C2—H2A	110.1 (8)	C8—C7—H7A	112.0 (8)
N1—C2—H2B	108.5 (8)	N1—C7—H7B	106.3 (8)
C3—C2—H2B	111.0 (8)	C8—C7—H7B	109.8 (8)
H2A—C2—H2B	106.7 (11)	H7A—C7—H7B	104.5 (11)
C2—C3—C4	110.49 (11)	C11—C8—C9	119.77 (11)
C2—C3—H3A	110.2 (9)	C11—C8—C7	118.88 (11)
C4—C3—H3A	110.8 (9)	C9—C8—C7	121.33 (11)
C2—C3—H3B	109.0 (9)	C11^{i}—C9—C8	119.79 (11)
C4—C3—H3B	108.9 (9)	C11^{i}—C9—C10	118.48 (11)
H3A—C3—H3B	107.5 (13)	C8—C9—C10	121.73 (11)
C3—C4—C5	109.63 (12)	C9—C10—H10A	111.6 (14)
C3—C4—H4A	109.9 (10)	C9—C10—H10B	112.8 (15)
C5—C4—H4A	107.2 (10)	H10A—C10—H10B	104.8 (19)
C3—C4—H4B	111.5 (10)	C9—C10—H10C	112.3 (14)
C5—C4—H4B	109.5 (10)	H10A—C10—H10C	106.1 (19)
H4A—C4—H4B	109.0 (14)	H10B—C10—H10C	108.6 (19)
C6—C5—C4	110.83 (12)	C9^{i}—C11—C8	120.44 (11)
C6—C5—H5A	109.3 (9)	C9^{i}—C11—C12	118.84 (11)
C4—C5—H5A	109.0 (9)	C8—C11—C12	120.72 (11)
C6—C5—H5B	110.2 (10)	C11—C12—H12A	113.1 (10)
C4—C5—H5B	111.0 (10)	C11—C12—H12B	113.0 (12)
H5A—C5—H5B	106.5 (13)	H12A—C12—H12B	107.0 (15)

N1—C6—C5	111.19 (11)	C11—C12—H12C	111.5 (11)
N1—C6—H6A	108.6 (9)	H12A—C12—H12C	104.8 (15)
C5—C6—H6A	110.8 (9)	H12B—C12—H12C	106.8 (16)
C6—N1—C2—C3	59.72 (14)	N1—C7—C8—C11	74.54 (14)
C7—N1—C2—C3	−178.35 (10)	N1—C7—C8—C9	−106.87 (13)
N1—C2—C3—C4	−57.54 (15)	C11—C8—C9—C11^{i}	0.46 (18)
C2—C3—C4—C5	53.97 (16)	C7—C8—C9—C11^{i}	−178.11 (10)
C3—C4—C5—C6	−54.05 (17)	C11—C8—C9—C10	−178.56 (11)
C2—N1—C6—C5	−59.38 (15)	C7—C8—C9—C10	2.87 (18)
C7—N1—C6—C5	177.78 (12)	C9—C8—C11—C9^{i}	−0.46 (18)
C4—C5—C6—N1	57.25 (16)	C7—C8—C11—C9^{i}	178.14 (10)
C6—N1—C7—C8	−170.47 (11)	C9—C8—C11—C12	179.05 (11)
C2—N1—C7—C8	67.37 (14)	C7—C8—C11—C12	−2.34 (17)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12*B*···N1	0.94 (2)	2.59 (2)	3.2204 (18)	124.8 (14)