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1,1′-(*p*-Phenylenedimethylene)-dipiperidin-4-one

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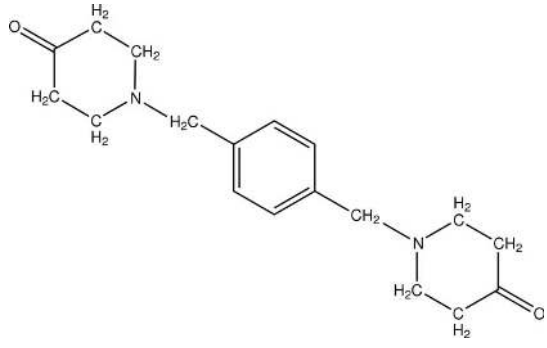
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.123; data-to-parameter ratio = 18.3.

In the molecule of the title compound, $C_{18}H_{24}N_2O_2$, the piperidine rings are in chair conformations. The crystal structure is stabilized by intermolecular C—H···O hydrogen bonding. There are neither C—H···π nor π–π interactions in the structure.

Related literature

For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For ring puckering parameters, see Cremer & Pople (1975).



Experimental

Crystal data

$C_{18}H_{24}N_2O_2$
M_r = 300.39
Monoclinic, $P2_1/n$
a = 6.2701 (5) Å
b = 8.0990 (6) Å
c = 15.8978 (13) Å
β = 98.275 (2)°
V = 798.91 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 K
0.19 × 0.17 × 0.15 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.984, T_{max} = 0.987
4782 measured reflections
1826 independent reflections
1424 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.123
S = 1.05
1826 reflections
100 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7B···O1^{i}	0.97	2.56	3.2235 (17)	126

Symmetry code: (i) $x, y-1, z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5127).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N. L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (1998). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). J. Am. Chem. Soc. *97*, 1354–1358.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2010). E**66**, o170 [doi:10.1107/S1600536809052908]

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S1. Comment

The configuration and conformation of the title compound, (I) and the atom numbering scheme are shown in the *ORTEP* drawing (Fig. 1). The piperidone ring exibits chair conformation as evident from the puckering parameters (Q)=0.549 (1) Å, θ = 173.4 (2) °, ψ = 181.9 (1) ° (Cremer & Pople, 1975).

In the crystal structure, an intermolecular C—H···O bond is found generating R22(24) motif (Bernstein *et al.*, 1995).

S2. Experimental

A mixture of 4-piperidone monohydrate hydrochloride (2 mol), 1,4-bis(bromomethyl)benzene (1 mol) and potassium carbonate (6 mol) in anhydrous benzene was refluxed for 7 h. The completion of reaction was monitored by TLC. Potassium carbonate was filtered off and the excess solvent was removed under reduced pressure. The solid obtained was purified over a column of silica gel (60–120 mesh size) using benzene-ethyl acetate (60–80 °C) in the ratio of 20:80. Yield: 40% m.p. 289°C.

S3. Refinement

The H atoms were placed in calculated positions and allowed to ride on their carrier atoms with C—H = 0.93–0.97 Å.U_{iso} = 1.2U_{eq}(C) for CH and CH_2 groups.

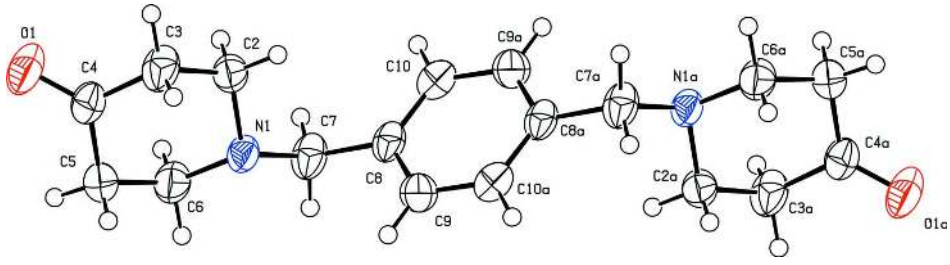

Figure 1

The molecular structure of title compound with atom numbering scheme and 50% probability displacement ellipsoids.

1,1′-(*p*-Phenylenedimethylene)dipiperidin-4-one

Crystal data

$C_{18}H_{24}N_2O_2$
M_r = 300.39
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 6.2701 (5) Å
b = 8.0990 (6) Å
c = 15.8978 (13) Å
β = 98.275 (2)°
V = 798.91 (11) Å^3
Z = 2
F(000) = 324
D_x = 1.249 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2500 reflections

θ = 2–30°
μ = 0.08 mm^{-1}
T = 293 K

Block, colourless
0.19 × 0.17 × 0.15 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.984, T_{max} = 0.987

4782 measured reflections
1826 independent reflections
1424 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 27.5°, θ_{min} = 2.6°
h = −8→4
k = −10→10
l = −20→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.123
S = 1.05
1826 reflections
100 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0639P)^2 + 0.1074P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Special details

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

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 $F^2 > \sigma(F^2)$ 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}
C2	0.2931 (2)	0.43945 (15)	0.38729 (9)	0.0451 (3)
H2A	0.3278	0.4330	0.3299	0.054*
H2B	0.4196	0.4060	0.4260	0.054*
C3	0.2358 (2)	0.61783 (16)	0.40660 (10)	0.0544 (4)
H3A	0.2191	0.6276	0.4661	0.065*
H3B	0.3519	0.6905	0.3959	0.065*
C4	0.0312 (2)	0.66908 (16)	0.35253 (8)	0.0440 (3)
C5	−0.1488 (2)	0.54905 (18)	0.35354 (11)	0.0584 (4)
H5A	−0.2680	0.5796	0.3105	0.070*
H5B	−0.1991	0.5528	0.4084	0.070*
C6	−0.0757 (2)	0.37490 (18)	0.33662 (10)	0.0544 (4)
H6A	−0.1918	0.2982	0.3419	0.065*
H6B	−0.0434	0.3682	0.2789	0.065*

C7	0.1742 (2)	0.15730 (16)	0.37561 (9)	0.0493 (4)
H7A	0.2250	0.1575	0.3208	0.059*
H7B	0.0458	0.0891	0.3705	0.059*
C8	0.3452 (2)	0.07991 (14)	0.44028 (8)	0.0405 (3)
C9	0.3083 (2)	0.05404 (16)	0.52331 (8)	0.0450 (3)
H9	0.1799	0.0900	0.5400	0.054*
C10	0.5395 (2)	0.02482 (16)	0.41824 (8)	0.0444 (3)
H10	0.5682	0.0411	0.3631	0.053*
N1	0.11571 (16)	0.32682 (12)	0.39582 (7)	0.0395 (3)
O1	0.01374 (19)	0.79483 (13)	0.31091 (7)	0.0657 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0372 (6)	0.0349 (7)	0.0605 (8)	−0.0033 (5)	−0.0021 (5)	0.0041 (6)
C3	0.0595 (8)	0.0329 (7)	0.0659 (9)	−0.0062 (6)	−0.0079 (7)	0.0018 (6)
C4	0.0549 (8)	0.0322 (6)	0.0448 (7)	0.0048 (5)	0.0072 (6)	0.0015 (5)
C5	0.0415 (7)	0.0509 (9)	0.0821 (10)	0.0057 (6)	0.0064 (7)	0.0227 (7)
C6	0.0423 (7)	0.0422 (8)	0.0731 (10)	−0.0085 (6)	−0.0107 (7)	0.0130 (7)
C7	0.0586 (8)	0.0308 (7)	0.0540 (8)	−0.0024 (6)	−0.0075 (6)	−0.0006 (6)
C8	0.0502 (7)	0.0233 (5)	0.0464 (7)	−0.0019 (5)	0.0015 (5)	−0.0002 (5)
C9	0.0462 (7)	0.0374 (7)	0.0528 (8)	0.0024 (5)	0.0113 (6)	−0.0021 (5)
C10	0.0573 (8)	0.0368 (7)	0.0401 (6)	−0.0039 (6)	0.0106 (6)	0.0016 (5)
N1	0.0389 (5)	0.0288 (5)	0.0484 (6)	−0.0026 (4)	−0.0022 (4)	0.0059 (4)
O1	0.0827 (8)	0.0385 (6)	0.0723 (7)	−0.0004 (5)	−0.0012 (6)	0.0175 (5)

Geometric parameters (Å, º)

C2—N1	1.4598 (16)	C6—H6A	0.9700
C2—C3	1.5304 (18)	C6—H6B	0.9700
C2—H2A	0.9700	C7—N1	1.4685 (17)
C2—H2B	0.9700	C7—C8	1.5104 (18)
C3—C4	1.4964 (19)	C7—H7A	0.9700
C3—H3A	0.9700	C7—H7B	0.9700
C3—H3B	0.9700	C8—C9	1.3884 (18)
C4—O1	1.2109 (16)	C8—C10	1.3888 (19)
C4—C5	1.492 (2)	C9—C10^{i}	1.3882 (18)
C5—C6	1.519 (2)	C9—H9	0.9300
C5—H5A	0.9700	C10—C9^{i}	1.3882 (18)
C5—H5B	0.9700	C10—H10	0.9300
C6—N1	1.4670 (16)		
N1—C2—C3	111.55 (11)	C5—C6—H6A	109.2
N1—C2—H2A	109.3	N1—C6—H6B	109.2
C3—C2—H2A	109.3	C5—C6—H6B	109.2
N1—C2—H2B	109.3	H6A—C6—H6B	107.9
C3—C2—H2B	109.3	N1—C7—C8	114.47 (10)
H2A—C2—H2B	108.0	N1—C7—H7A	108.6

C4—C3—C2	110.67 (11)	C8—C7—H7A	108.6
C4—C3—H3A	109.5	N1—C7—H7B	108.6
C2—C3—H3A	109.5	C8—C7—H7B	108.6
C4—C3—H3B	109.5	H7A—C7—H7B	107.6
C2—C3—H3B	109.5	C9—C8—C10	117.61 (11)
H3A—C3—H3B	108.1	C9—C8—C7	120.72 (12)
O1—C4—C5	123.01 (13)	C10—C8—C7	121.59 (12)
O1—C4—C3	123.37 (13)	C10^{i}—C9—C8	120.89 (12)
C5—C4—C3	113.62 (11)	C10^{i}—C9—H9	119.6
C4—C5—C6	110.77 (12)	C8—C9—H9	119.6
C4—C5—H5A	109.5	C9^{i}—C10—C8	121.50 (12)
C6—C5—H5A	109.5	C9^{i}—C10—H10	119.3
C4—C5—H5B	109.5	C8—C10—H10	119.3
C6—C5—H5B	109.5	C2—N1—C6	109.77 (10)
H5A—C5—H5B	108.1	C2—N1—C7	110.25 (11)
N1—C6—C5	111.90 (12)	C6—N1—C7	108.36 (10)
N1—C6—H6A	109.2		

N1—C2—C3—C4	−54.57 (16)	C7—C8—C9—C10^{i}	176.62 (11)
C2—C3—C4—O1	−129.37 (15)	C9—C8—C10—C9^{i}	0.3 (2)
C2—C3—C4—C5	49.69 (17)	C7—C8—C10—C9^{i}	−176.59 (12)
O1—C4—C5—C6	129.33 (15)	C3—C2—N1—C6	59.82 (15)
C3—C4—C5—C6	−49.73 (18)	C3—C2—N1—C7	179.12 (11)
C4—C5—C6—N1	54.57 (17)	C5—C6—N1—C2	−60.02 (16)
N1—C7—C8—C9	62.64 (17)	C5—C6—N1—C7	179.53 (12)
N1—C7—C8—C10	−120.58 (14)	C8—C7—N1—C2	69.70 (15)
C10—C8—C9—C10^{i}	−0.3 (2)	C8—C7—N1—C6	−170.15 (12)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*B*···O1^{ii}	0.97	2.56	3.2235 (17)	126

Symmetry code: (ii) *x*, *y*−1, *z*.