organic compounds

3-[(E)-4-Methoxybenzylidene]-1-methyl-piperidin-4-one

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The piperidone ring of the title compound, $C_{14}H_{17}NO_2$, adopts a half-chair conformation. The crystal packing is stabilized by intermolecular C—H···O interactions, which generate a $C(8)$ chain running along the b axis.

Related literature

For related literature, see: Abignente & Biniecka-Picazio (1977); Angle & Breitenbucher (1995); Cremer & Pople (1975); Nardelli (1983); Wang & Wuorola (1992).
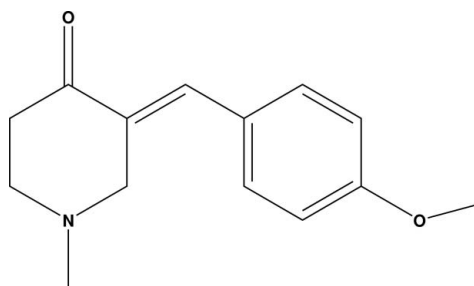
Experimental

Crystal data

$C_{14}H_{17}NO_2$	Orthorhombic, $P2_12_12_1$
$M_r = 231.29$	$a = 7.5212$ (7) Å
$b = 12.4097$ (11) Å	Mo $K\alpha$ radiation
$c = 13.5062$ (12) Å	$\mu = 0.08$ mm^{-1}
$V = 1260.6$ (2) Å^3	$T = 293$ (2) K
$Z = 4$	$0.24 \times 0.22 \times 0.21$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	1729 independent reflections
Absorption correction: none	1577 reflections with $I > 2\sigma(I)$
10874 measured reflections	$R_{int} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	156 parameters
$wR(F^2) = 0.128$	H-atom parameters constrained
$S = 1.11$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
1729 reflections	$\Delta\rho_{min} = -0.13$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10···O1^i	0.93	2.54	3.419 (3)	157

Symmetry code: (i) $-x, y + \frac{1}{2}, -z + \frac{3}{2}$.

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, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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supplementary materials

3-[(*E*)-4-Methoxybenzylidene]-1-methylpiperidin-4-one

D. Gayathri, D. Velmurugan, R. R. Kumar, S. Perumal and K. Ravikumar

Comment

Substituted 4-piperidones are important synthetic intermediates for the preparation of various pharmaceuticals (Wang & Wuorola, 1992). 4-Piperidones are also widely prevalent in natural products such as alkaloids (Angle & Breitenbucher, 1995). Derivatives of 4-piperidones have been found to exhibit spasmolytic activities (Abignente & Biniecka-Picazio, 1977). Since, the title compound is pharmacologically important, the crystal structure of the title compound has been determined by X-ray diffraction.

The sum of the bond angles around N1 [331.4 (6)°] indicate the sp^3 hybridization. The torsion angles around C10—C11—O2—C14 [179.4 (2)°] and C12—C11—O2—C14 [−0.8 (3)°] indicate that the methoxy group is planar with the phenyl ring.

The piperidone ring adopts a half-chair conformation with the puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) being $q_2 = 0.366$ (3) Å, $q_3 = 0.361$ (3) Å, $Q_T = 0.515$ (3)Å and $\theta = 45.4$ (3)°. The molecular conformation is stabilized by weak C—H⋯O intramolecular interactions. The crystal packing is stabilized by C—H⋯O intermolecular interactions generating a chain C(8) running along *b* axis.

Experimental

A mixture of 1-methyl-4-piperidone (1 mmol) and pyrrolidine (1.2 mmol) was taken in a glass tube, mixed well and kept aside for 5 min at ambient temperature. To this mixture, 4-methoxybenzaldehyde (1 mmol) was added, mixed thoroughly and the tube containing the mixture was partially immersed in a silica bath placed in a microwave oven and irradiated at 4 power level for 8 minutes. The progress of the reaction was monitored after every 1 min of irradiation by TLC with petroleum ether:ethyl acetate (1:2 *v/v* mixture) as eluent. After each irradiation, the reaction mixture was cooled to room temperature and mixed well. The maximum temperature of the silica bath, measured immediately after each irradiation was over by stirring the silica bath with the thermometer, was found to be 65 °C. After completion of the reaction as evident from the TLC, the product was purified by column chromatography using petroleum ether:ethyl acetate (7:2 *v/v*) mixture and crystallized from ethyl acetate.

Refinement

In the absence of anomalous scatterers Friedel pairs had been merged prior to refinement. All H-atoms were refined using a riding model with d(C—H) = 0.93 Å, $U_{iso}=1.2U_{eq}$ (C) for aromatic C atoms, 0.97 Å, $U_{iso} = 1.2U_{eq}$ (C) for methylene and 0.96 Å, $U_{iso} = 1.5U_{eq}$ (C) for methyl groups.
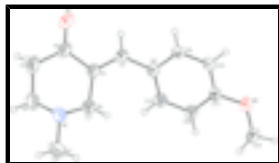
supplementary materials

Figures

Fig. 1. The molecular structure of title compound, showing 30% probability displacement ellipsoids.
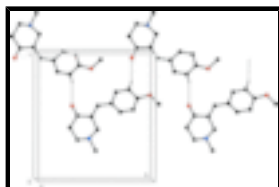

Fig. 2. The molecular packing of the title compound, viewed down the *a* axis.

3-[(*E*)-4-Methoxybenzylidene]-1-methylpiperidin-4-one

Crystal data

$C_{14}H_{17}NO_2$	$F_{000} = 496$
$M_r = 231.29$	$D_x = 1.219$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 861 reflections
$a = 7.5212 (7)$ Å	$\theta = 2.2–25.0°$
$b = 12.4097 (11)$ Å	$\mu = 0.08$ mm^{-1}
$c = 13.5062 (12)$ Å	$T = 293 (2)$ K
$V = 1260.6 (2)$ Å^3	Block, pale yellow
$Z = 4$	$0.24 \times 0.22 \times 0.21$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	1577 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.019$
Monochromator: graphite	$\theta_{max} = 28.1°$
$T = 293(2)$ K	$\theta_{min} = 2.2°$
ω scans	$h = -9 \rightarrow 9$
Absorption correction: none	$k = -16 \rightarrow 16$
10874 measured reflections	$l = -17 \rightarrow 17$
1729 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.045$	$w = 1/[\sigma^2(F_o^2) + (0.083P)^2 + 0.0611P]$ where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2) = 0.128$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.11$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
1729 reflections	$\Delta\rho_{min} = -0.13$ e Å^{-3}
156 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure:
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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	$U_{iso}*/U_{eq}$
C1	−0.1137 (5)	0.0252 (2)	1.28219 (18)	0.0864 (8)
H1A	−0.0039	0.0433	1.3144	0.130*
H1B	−0.1816	−0.0218	1.3241	0.130*
H1C	−0.1803	0.0898	1.2697	0.130*
C2	0.0338 (4)	−0.1243 (2)	1.20225 (17)	0.0802 (7)
H2A	−0.0149	−0.1685	1.2549	0.096*
H2B	0.1533	−0.1031	1.2210	0.096*
C3	0.0400 (5)	−0.18853 (18)	1.1072 (2)	0.0845 (7)
H3A	0.1321	−0.2429	1.1131	0.101*
H3B	−0.0725	−0.2257	1.0992	0.101*
C4	0.0749 (4)	−0.12308 (16)	1.01586 (16)	0.0697 (6)
C5	0.0494 (3)	−0.00418 (14)	1.02172 (14)	0.0548 (5)
C6	0.0120 (3)	0.04524 (15)	1.12177 (14)	0.0558 (4)
H6A	0.1232	0.0685	1.1512	0.067*
H6B	−0.0622	0.1085	1.1130	0.067*
C7	0.0594 (3)	0.05043 (14)	0.93680 (15)	0.0560 (5)
H7	0.0790	0.0090	0.8805	0.067*
C8	0.0436 (3)	0.16662 (14)	0.91996 (13)	0.0510 (4)
C9	−0.0213 (3)	0.20203 (14)	0.82852 (13)	0.0562 (5)
H9	−0.0506	0.1515	0.7804	0.067*
C10	−0.0427 (3)	0.30978 (15)	0.80817 (12)	0.0576 (5)
H10	−0.0879	0.3314	0.7472	0.069*
C11	0.0031 (2)	0.38597 (14)	0.87840 (13)	0.0511 (4)
C12	0.0740 (3)	0.35353 (14)	0.96828 (14)	0.0585 (5)
H12	0.1079	0.4045	1.0151	0.070*

C13	0.0938 (3)	0.24434 (17)	0.98781 (15)	0.0581 (5)
H13	0.1422	0.2229	1.0481	0.070*
C14	0.0194 (4)	0.57070 (15)	0.92477 (17)	0.0667 (6)
H14A	−0.0449	0.5570	0.9849	0.100*
H14B	−0.0116	0.6407	0.8999	0.100*
H14C	0.1448	0.5681	0.9379	0.100*
N1	−0.0762 (3)	−0.02883 (15)	1.18885 (13)	0.0656 (5)
O1	0.1230 (4)	−0.16737 (12)	0.94008 (14)	0.0993 (7)
O2	−0.0251 (2)	0.49113 (10)	0.85323 (10)	0.0627 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.105 (2)	0.0972 (18)	0.0569 (12)	−0.0171 (16)	0.0036 (13)	0.0011 (12)
C2	0.1040 (18)	0.0634 (11)	0.0731 (13)	−0.0072 (14)	−0.0128 (14)	0.0222 (11)
C3	0.1134 (19)	0.0459 (9)	0.0942 (16)	−0.0027 (13)	−0.0080 (17)	0.0165 (11)
C4	0.0965 (15)	0.0404 (8)	0.0723 (12)	0.0031 (10)	−0.0171 (13)	−0.0023 (9)
C5	0.0662 (11)	0.0391 (7)	0.0590 (10)	0.0011 (8)	−0.0092 (9)	−0.0002 (7)
C6	0.0660 (11)	0.0464 (8)	0.0551 (9)	−0.0030 (8)	−0.0093 (9)	0.0009 (7)
C7	0.0701 (11)	0.0430 (8)	0.0551 (9)	0.0031 (8)	−0.0019 (9)	−0.0053 (7)
C8	0.0603 (10)	0.0433 (8)	0.0493 (8)	0.0005 (8)	0.0021 (8)	0.0001 (6)
C9	0.0787 (12)	0.0476 (8)	0.0423 (7)	−0.0009 (9)	0.0058 (9)	−0.0040 (6)
C10	0.0793 (12)	0.0538 (9)	0.0397 (7)	0.0015 (10)	0.0022 (9)	0.0058 (7)
C11	0.0602 (9)	0.0421 (7)	0.0510 (8)	−0.0017 (8)	0.0055 (8)	0.0055 (7)
C12	0.0744 (11)	0.0443 (9)	0.0568 (10)	−0.0085 (9)	−0.0103 (9)	−0.0004 (7)
C13	0.0713 (11)	0.0488 (9)	0.0544 (9)	−0.0029 (9)	−0.0150 (9)	0.0057 (7)
C14	0.0878 (14)	0.0414 (8)	0.0708 (12)	−0.0001 (10)	0.0100 (12)	−0.0005 (8)
N1	0.0784 (11)	0.0618 (9)	0.0565 (8)	−0.0120 (9)	−0.0084 (9)	0.0061 (7)
O1	0.169 (2)	0.0465 (7)	0.0828 (11)	0.0139 (12)	−0.0067 (13)	−0.0083 (8)
O2	0.0866 (10)	0.0415 (6)	0.0600 (7)	−0.0016 (7)	−0.0008 (8)	0.0077 (5)

Geometric parameters (Å, °)

C1—N1	1.456 (3)	C7—C8	1.465 (2)
C1—H1A	0.9600	C7—H7	0.9300
C1—H1B	0.9600	C8—C13	1.383 (3)
C1—H1C	0.9600	C8—C9	1.399 (3)
C2—N1	1.457 (3)	C9—C10	1.374 (3)
C2—C3	1.511 (4)	C9—H9	0.9300
C2—H2A	0.9700	C10—C11	1.383 (3)
C2—H2B	0.9700	C10—H10	0.9300
C3—C4	1.500 (3)	C11—O2	1.365 (2)
C3—H3A	0.9700	C11—C12	1.385 (3)
C3—H3B	0.9700	C12—C13	1.389 (3)
C4—O1	1.217 (3)	C12—H12	0.9300
C4—C5	1.490 (3)	C13—H13	0.9300
C5—C7	1.334 (3)	C14—O2	1.421 (3)
C5—C6	1.510 (3)	C14—H14A	0.9600
C6—N1	1.451 (3)	C14—H14B	0.9600

C6—H6A	0.9700	C14—H14C	0.9600
C6—H6B	0.9700		
N1—C1—H1A	109.5	C5—C7—H7	115.5
N1—C1—H1B	109.5	C8—C7—H7	115.5
H1A—C1—H1B	109.5	C13—C8—C9	117.47 (16)
N1—C1—H1C	109.5	C13—C8—C7	124.17 (18)
H1A—C1—H1C	109.5	C9—C8—C7	118.33 (16)
H1B—C1—H1C	109.5	C10—C9—C8	121.53 (17)
N1—C2—C3	109.93 (19)	C10—C9—H9	119.2
N1—C2—H2A	109.7	C8—C9—H9	119.2
C3—C2—H2A	109.7	C9—C10—C11	119.92 (17)
N1—C2—H2B	109.7	C9—C10—H10	120.0
C3—C2—H2B	109.7	C11—C10—H10	120.0
H2A—C2—H2B	108.2	O2—C11—C12	123.75 (16)
C4—C3—C2	114.74 (18)	O2—C11—C10	116.37 (16)
C4—C3—H3A	108.6	C12—C11—C10	119.88 (16)
C2—C3—H3A	108.6	C11—C12—C13	119.43 (17)
C4—C3—H3B	108.6	C11—C12—H12	120.3
C2—C3—H3B	108.6	C13—C12—H12	120.3
H3A—C3—H3B	107.6	C8—C13—C12	121.69 (18)
O1—C4—C5	122.0 (2)	C8—C13—H13	119.2
O1—C4—C3	119.95 (19)	C12—C13—H13	119.2
C5—C4—C3	118.0 (2)	O2—C14—H14A	109.5
C7—C5—C4	116.75 (17)	O2—C14—H14B	109.5
C7—C5—C6	124.98 (16)	H14A—C14—H14B	109.5
C4—C5—C6	118.27 (16)	O2—C14—H14C	109.5
N1—C6—C5	112.74 (16)	H14A—C14—H14C	109.5
N1—C6—H6A	109.0	H14B—C14—H14C	109.5
C5—C6—H6A	109.0	C6—N1—C1	109.72 (18)
N1—C6—H6B	109.0	C6—N1—C2	109.5 (2)
C5—C6—H6B	109.0	C1—N1—C2	112.18 (19)
H6A—C6—H6B	107.8	C11—O2—C14	117.27 (15)
C5—C7—C8	128.98 (17)		
N1—C2—C3—C4	46.7 (3)	C8—C9—C10—C11	1.1 (3)
C2—C3—C4—O1	163.0 (3)	C9—C10—C11—O2	−178.8 (2)
C2—C3—C4—C5	−16.8 (4)	C9—C10—C11—C12	1.3 (3)
O1—C4—C5—C7	8.4 (4)	O2—C11—C12—C13	178.5 (2)
C3—C4—C5—C7	−171.8 (2)	C10—C11—C12—C13	−1.7 (3)
O1—C4—C5—C6	−172.5 (3)	C9—C8—C13—C12	2.7 (3)
C3—C4—C5—C6	7.3 (3)	C7—C8—C13—C12	−179.2 (2)
C7—C5—C6—N1	151.3 (2)	C11—C12—C13—C8	−0.4 (3)
C4—C5—C6—N1	−27.8 (3)	C5—C6—N1—C1	−178.0 (2)
C4—C5—C7—C8	−178.6 (2)	C5—C6—N1—C2	58.5 (2)
C6—C5—C7—C8	2.3 (4)	C3—C2—N1—C6	−68.5 (3)
C5—C7—C8—C13	30.6 (4)	C3—C2—N1—C1	169.4 (2)
C5—C7—C8—C9	−151.3 (2)	C12—C11—O2—C14	−0.8 (3)
C13—C8—C9—C10	−3.1 (3)	C10—C11—O2—C14	179.37 (19)
C7—C8—C9—C10	178.8 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O1[i]	0.93	2.54	3.419 (3)	157

Symmetry codes: (i) −*x*, *y*+1/2, −*z*+3/2.

Fig. 1

Fig. 2