Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,6-Bis(3-methoxyphenyl)-3-methylpiperidin-4-one

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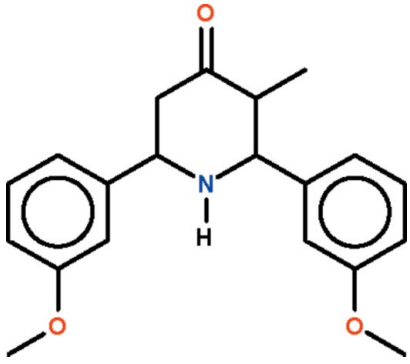
Received 27 October 2009; accepted 29 October 2009

Key indicators: single-crystal X-ray study; T = 290 K; mean σ(C–C) = 0.004 Å; R factor = 0.063; wR factor = 0.154; data-to-parameter ratio = 14.1.

In the molecule of the title compound, $C_{20}H_{23}NO_3$, the bulky methoxyphenyl substituents at the equatorial 2,6-positions crowd the vicinity of the equatorial amino H atom and prevent it from forming intermolecular hydrogen bonds. The piperidine ring adopts a distorted chair conformation.

Related literature

For the crystal structure of a related piperidinone compound, see: Nithya *et al.* (2009).



Experimental

Crystal data

$C_{20}H_{23}NO_3$
M_r = 325.39
Monoclinic, $C2/c$
a = 28.695 (3) Å
b = 10.9717 (12) Å
c = 11.3946 (13) Å
β = 95.078 (2)°
V = 3573.3 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 290 K
0.35 × 0.12 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
12581 measured reflections
3148 independent reflections
1751 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.154
S = 1.04
3148 reflections
224 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

The authors thank the Department of Science and Technology, India, for use of the diffraction facility set up under the IRHPA–DST programme at IISc. FNK thanks the DST for Fast Track Proposal funding. The authors also thank VIT University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2955).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
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supporting information

Acta Cryst. (2009). E**65**, o2984 [doi:10.1107/S1600536809045346]

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S1. Experimental

Ammonium acetate (1 mmol), *m*-methoxybenzaldehyde (2 mmol) and ethylmethyl ketone (1 mmol) was heated until the colour of the solution turned yellow. After the completion of the reaction (as monitored by TLC), the product was dissolved in ether (10 ml). The solution was treated with aqueous hydrochloric acid [20 ml, 1:1 (*v*/*v*)]. The hydrochloride salt of the piperidin-4-one was collected and washed with ether. The base was liberated from an alcohol solution of the hydrochloride by the addition of a slight excess of aqueous ammonia at 273 K. The product was collected and recrystallized from ethanol.

S2. Refinement

C-bound H-atoms were placed in calculated positions (C-H = 0.93–0.98 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2–1.5U_{eq}(C).

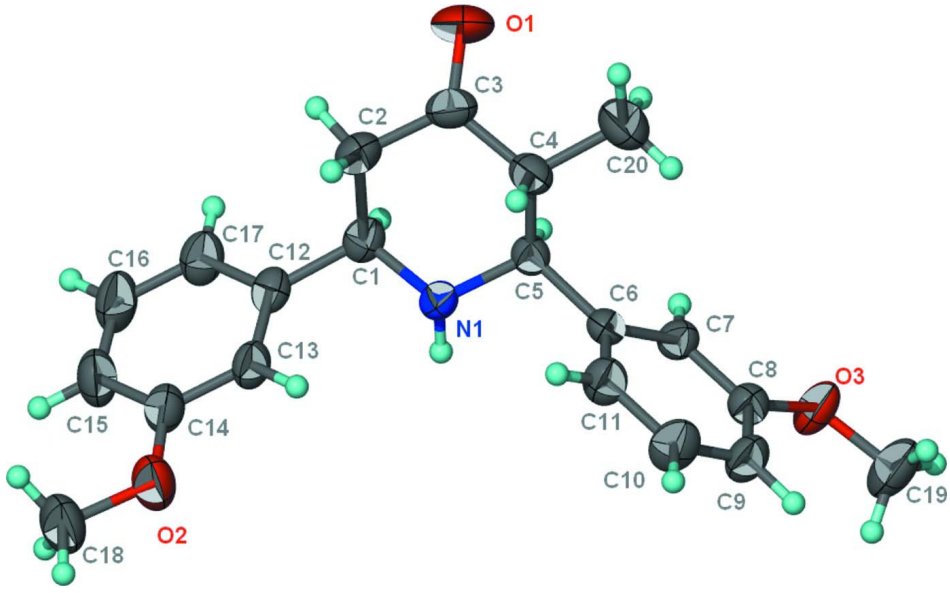

Figure 1

Displacement ellipsoid plot (Barbour, 2001) of $C_{20}H_{23}NO_3$ at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

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Crystal data

$C_{20}H_{23}NO_3$
$M_r = 325.39$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 28.695$ (3) Å
$b = 10.9717$ (12) Å
$c = 11.3946$ (13) Å
$\beta = 95.078$ (2)°
$V = 3573.3$ (7) Å^3
$Z = 8$
$F(000) = 1392$
$D_x = 1.210$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1065 reflections
$\theta = 2.0$–$20.8°$
$\mu = 0.08$ mm^{-1}
$T = 290$ K
Plate, colourless
0.35 × 0.12 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
12581 measured reflections
3148 independent reflections
1751 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.4°$
$h = -33 \rightarrow 34$
$k = -13 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.154$
$S = 1.04$
3148 reflections
224 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0658P)^2 + 0.6442P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.58419 (9)	0.8518 (2)	0.9145 (2)	0.1002 (10)
O2	0.76371 (8)	0.5426 (2)	0.51929 (18)	0.0750 (7)
O3	0.57121 (8)	0.17943 (18)	1.10361 (17)	0.0647 (6)
N1	0.65515 (8)	0.5548 (2)	0.83450 (19)	0.0438 (6)
H1N	0.6737 (9)	0.493 (2)	0.850 (2)	0.042 (8)*
C1	0.68070 (10)	0.6693 (3)	0.8526 (2)	0.0460 (7)
H1	0.6910	0.6772	0.9366	0.055*
C2	0.64767 (11)	0.7743 (3)	0.8172 (3)	0.0585 (9)
H2A	0.6627	0.8505	0.8419	0.070*
H2B	0.6420	0.7760	0.7320	0.070*
C3	0.60185 (12)	0.7651 (3)	0.8696 (3)	0.0588 (9)
C4	0.57864 (10)	0.6416 (3)	0.8627 (2)	0.0488 (8)
H4	0.5697	0.6251	0.7792	0.059*

C5	0.61533 (9)	0.5452 (2)	0.9056 (2)	0.0405 (7)
H5	0.6262	0.5630	0.9878	0.049*
C6	0.59593 (9)	0.4172 (2)	0.8989 (2)	0.0392 (7)
C7	0.59208 (9)	0.3507 (2)	1.0002 (2)	0.0429 (7)
H7	0.6019	0.3850	1.0729	0.051*
C8	0.57388 (10)	0.2338 (3)	0.9957 (2)	0.0456 (7)
C9	0.56020 (11)	0.1801 (3)	0.8898 (3)	0.0573 (8)
H9	0.5487	0.1009	0.8864	0.069*
C10	0.56400 (11)	0.2471 (3)	0.7879 (3)	0.0646 (9)
H10	0.5544	0.2123	0.7153	0.078*
C11	0.58152 (10)	0.3632 (3)	0.7912 (2)	0.0525 (8)
H11	0.5838	0.4061	0.7216	0.063*
C12	0.72330 (10)	0.6733 (3)	0.7837 (2)	0.0445 (7)
C13	0.72600 (10)	0.6065 (3)	0.6817 (2)	0.0500 (8)
H13	0.7013	0.5558	0.6550	0.060*
C14	0.76509 (11)	0.6143 (3)	0.6189 (3)	0.0549 (8)
C15	0.80184 (12)	0.6882 (3)	0.6568 (3)	0.0686 (10)
H15	0.8284	0.6922	0.6155	0.082*
C16	0.79871 (12)	0.7568 (3)	0.7577 (3)	0.0716 (10)
H16	0.8232	0.8085	0.7834	0.086*
C17	0.76009 (11)	0.7498 (3)	0.8206 (3)	0.0597 (9)
H17	0.7587	0.7967	0.8882	0.072*
C18	0.80099 (13)	0.5566 (3)	0.4451 (3)	0.0848 (12)
H18A	0.8021	0.6396	0.4189	0.127*
H18B	0.7958	0.5036	0.3782	0.127*
H18C	0.8301	0.5359	0.4885	0.127*
C19	0.55104 (15)	0.0625 (3)	1.1084 (3)	0.0877 (12)
H19A	0.5192	0.0654	1.0746	0.132*
H19B	0.5518	0.0365	1.1890	0.132*
H19C	0.5684	0.0060	1.0650	0.132*
C20	0.53454 (12)	0.6374 (3)	0.9252 (3)	0.0826 (11)
H20A	0.5421	0.6511	1.0079	0.124*
H20B	0.5201	0.5589	0.9136	0.124*
H20C	0.5134	0.6994	0.8939	0.124*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.119 (2)	0.0536 (16)	0.135 (2)	0.0140 (15)	0.0511 (19)	−0.0246 (15)
O2	0.0798 (17)	0.0788 (16)	0.0713 (14)	−0.0245 (13)	0.0343 (13)	−0.0180 (13)
O3	0.0959 (17)	0.0445 (13)	0.0538 (13)	−0.0223 (12)	0.0074 (11)	0.0049 (10)
N1	0.0422 (15)	0.0342 (15)	0.0560 (15)	0.0009 (13)	0.0100 (12)	0.0041 (12)
C1	0.0515 (19)	0.0455 (18)	0.0403 (15)	−0.0075 (16)	0.0001 (14)	−0.0005 (13)
C2	0.069 (2)	0.0394 (19)	0.068 (2)	−0.0052 (16)	0.0137 (17)	−0.0052 (15)
C3	0.071 (2)	0.042 (2)	0.064 (2)	0.0102 (18)	0.0113 (17)	−0.0019 (15)
C4	0.0485 (19)	0.051 (2)	0.0485 (17)	0.0065 (16)	0.0123 (14)	−0.0005 (14)
C5	0.0421 (17)	0.0418 (17)	0.0381 (14)	−0.0016 (14)	0.0074 (12)	−0.0017 (12)
C6	0.0373 (16)	0.0382 (17)	0.0431 (16)	0.0028 (13)	0.0092 (13)	−0.0022 (13)

C7	0.0485 (18)	0.0393 (18)	0.0410 (16)	−0.0024 (14)	0.0049 (13)	−0.0036 (13)
C8	0.0482 (19)	0.0446 (19)	0.0447 (16)	−0.0016 (15)	0.0075 (13)	−0.0012 (14)
C9	0.068 (2)	0.0440 (19)	0.060 (2)	−0.0164 (16)	0.0096 (16)	−0.0051 (15)
C10	0.086 (3)	0.060 (2)	0.0469 (18)	−0.019 (2)	0.0026 (16)	−0.0138 (16)
C11	0.063 (2)	0.055 (2)	0.0407 (17)	−0.0082 (17)	0.0069 (14)	−0.0025 (14)
C12	0.0453 (18)	0.0438 (17)	0.0438 (16)	−0.0059 (15)	0.0006 (14)	0.0074 (13)
C13	0.0480 (19)	0.0487 (19)	0.0536 (18)	−0.0162 (15)	0.0067 (15)	−0.0026 (14)
C14	0.057 (2)	0.053 (2)	0.0560 (19)	−0.0118 (17)	0.0130 (16)	0.0019 (16)
C15	0.060 (2)	0.077 (2)	0.072 (2)	−0.019 (2)	0.0216 (18)	0.0035 (19)
C16	0.062 (2)	0.077 (2)	0.077 (2)	−0.032 (2)	0.0088 (19)	−0.002 (2)
C17	0.061 (2)	0.064 (2)	0.0535 (18)	−0.0187 (19)	0.0034 (16)	−0.0029 (16)
C18	0.089 (3)	0.094 (3)	0.077 (2)	−0.020 (2)	0.042 (2)	−0.012 (2)
C19	0.140 (4)	0.054 (2)	0.071 (2)	−0.031 (2)	0.021 (2)	0.0032 (18)
C20	0.074 (3)	0.076 (3)	0.103 (3)	0.017 (2)	0.039 (2)	0.011 (2)

Geometric parameters (Å, º)

O1—C3	1.212 (3)	C9—C10	1.388 (4)
O2—C14	1.379 (3)	C9—H9	0.93
O2—C18	1.429 (3)	C10—C11	1.368 (4)
O3—C8	1.375 (3)	C10—H10	0.93
O3—C19	1.411 (3)	C11—H11	0.93
N1—C1	1.460 (3)	C12—C13	1.382 (4)
N1—C5	1.462 (3)	C12—C17	1.384 (4)
N1—H1N	0.87 (3)	C13—C14	1.385 (4)
C1—C12	1.511 (4)	C13—H13	0.93
C1—C2	1.524 (4)	C14—C15	1.369 (4)
C1—H1	0.98	C15—C16	1.384 (4)
C2—C3	1.495 (4)	C15—H15	0.93
C2—H2A	0.97	C16—C17	1.374 (4)
C2—H2B	0.97	C16—H16	0.93
C3—C4	1.509 (4)	C17—H17	0.93
C4—C20	1.507 (4)	C18—H18A	0.96
C4—C5	1.541 (4)	C18—H18B	0.96
C4—H4	0.98	C18—H18C	0.96
C5—C6	1.511 (3)	C19—H19A	0.96
C5—H5	0.98	C19—H19B	0.96
C6—C7	1.379 (3)	C19—H19C	0.96
C6—C11	1.392 (3)	C20—H20A	0.96
C7—C8	1.384 (4)	C20—H20B	0.96
C7—H7	0.93	C20—H20C	0.96
C8—C9	1.368 (4)		
C14—O2—C18	117.2 (2)	C10—C9—H9	120.9
C8—O3—C19	119.0 (2)	C11—C10—C9	121.7 (3)
C1—N1—C5	113.0 (2)	C11—C10—H10	119.2
C1—N1—H1N	110.2 (17)	C9—C10—H10	119.2
C5—N1—H1N	109.0 (16)	C10—C11—C6	120.0 (3)

N1—C1—C12	111.8 (2)	C10—C11—H11	120.0
N1—C1—C2	108.7 (2)	C6—C11—H11	120.0
C12—C1—C2	110.7 (2)	C13—C12—C17	118.6 (3)
N1—C1—H1	108.5	C13—C12—C1	122.0 (3)
C12—C1—H1	108.5	C17—C12—C1	119.4 (3)
C2—C1—H1	108.5	C12—C13—C14	120.6 (3)
C3—C2—C1	113.0 (2)	C12—C13—H13	119.7
C3—C2—H2A	109.0	C14—C13—H13	119.7
C1—C2—H2A	109.0	C15—C14—O2	124.2 (3)
C3—C2—H2B	109.0	C15—C14—C13	120.7 (3)
C1—C2—H2B	109.0	O2—C14—C13	115.1 (3)
H2A—C2—H2B	107.8	C14—C15—C16	118.6 (3)
O1—C3—C2	122.1 (3)	C14—C15—H15	120.7
O1—C3—C4	121.8 (3)	C16—C15—H15	120.7
C2—C3—C4	116.1 (3)	C17—C16—C15	121.2 (3)
C20—C4—C3	112.9 (3)	C17—C16—H16	119.4
C20—C4—C5	114.0 (2)	C15—C16—H16	119.4
C3—C4—C5	108.3 (2)	C16—C17—C12	120.3 (3)
C20—C4—H4	107.1	C16—C17—H17	119.9
C3—C4—H4	107.1	C12—C17—H17	119.9
C5—C4—H4	107.1	O2—C18—H18A	109.5
N1—C5—C6	110.0 (2)	O2—C18—H18B	109.5
N1—C5—C4	108.7 (2)	H18A—C18—H18B	109.5
C6—C5—C4	112.6 (2)	O2—C18—H18C	109.5
N1—C5—H5	108.5	H18A—C18—H18C	109.5
C6—C5—H5	108.5	H18B—C18—H18C	109.5
C4—C5—H5	108.5	O3—C19—H19A	109.5
C7—C6—C11	118.3 (3)	O3—C19—H19B	109.5
C7—C6—C5	120.4 (2)	H19A—C19—H19B	109.5
C11—C6—C5	121.3 (2)	O3—C19—H19C	109.5
C6—C7—C8	121.1 (3)	H19A—C19—H19C	109.5
C6—C7—H7	119.4	H19B—C19—H19C	109.5
C8—C7—H7	119.4	C4—C20—H20A	109.5
C9—C8—O3	124.5 (3)	C4—C20—H20B	109.5
C9—C8—C7	120.7 (3)	H20A—C20—H20B	109.5
O3—C8—C7	114.8 (2)	C4—C20—H20C	109.5
C8—C9—C10	118.2 (3)	H20A—C20—H20C	109.5
C8—C9—H9	120.9	H20B—C20—H20C	109.5

C5—N1—C1—C12	176.5 (2)	C6—C7—C8—C9	1.5 (4)
C5—N1—C1—C2	−61.0 (3)	C6—C7—C8—O3	−178.7 (2)
N1—C1—C2—C3	48.7 (3)	O3—C8—C9—C10	178.6 (3)
C12—C1—C2—C3	171.8 (2)	C7—C8—C9—C10	−1.6 (4)
C1—C2—C3—O1	134.6 (3)	C8—C9—C10—C11	1.0 (5)
C1—C2—C3—C4	−45.8 (4)	C9—C10—C11—C6	−0.2 (5)
O1—C3—C4—C20	−4.6 (4)	C7—C6—C11—C10	0.1 (4)
C2—C3—C4—C20	175.7 (3)	C5—C6—C11—C10	−179.9 (3)
O1—C3—C4—C5	−131.9 (3)	N1—C1—C12—C13	26.1 (4)

C2—C3—C4—C5	48.5 (3)	C2—C1—C12—C13	−95.1 (3)
C1—N1—C5—C6	−170.0 (2)	N1—C1—C12—C17	−156.6 (3)
C1—N1—C5—C4	66.3 (3)	C2—C1—C12—C17	82.1 (3)
C20—C4—C5—N1	177.2 (3)	C17—C12—C13—C14	1.2 (4)
C3—C4—C5—N1	−56.3 (3)	C1—C12—C13—C14	178.5 (3)
C20—C4—C5—C6	55.1 (3)	C18—O2—C14—C15	−6.7 (5)
C3—C4—C5—C6	−178.3 (2)	C18—O2—C14—C13	173.8 (3)
N1—C5—C6—C7	124.1 (3)	C12—C13—C14—C15	0.1 (5)
C4—C5—C6—C7	−114.6 (3)	C12—C13—C14—O2	179.5 (3)
N1—C5—C6—C11	−56.0 (3)	O2—C14—C15—C16	179.3 (3)
C4—C5—C6—C11	65.4 (3)	C13—C14—C15—C16	−1.3 (5)
C11—C6—C7—C8	−0.7 (4)	C14—C15—C16—C17	1.2 (5)
C5—C6—C7—C8	179.2 (2)	C15—C16—C17—C12	0.0 (5)
C19—O3—C8—C9	−3.2 (4)	C13—C12—C17—C16	−1.2 (4)
C19—O3—C8—C7	177.0 (3)	C1—C12—C17—C16	−178.6 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*B*···O3^{i}	0.97	2.45	3.170 (4)	131
C19—H19*C*···O1^{ii}	0.96	2.48	3.392 (4)	159

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