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

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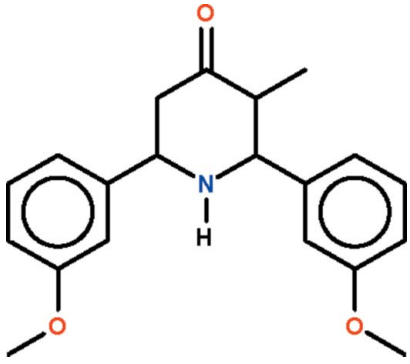
Key indicators: single-crystal X-ray study; T = 290 K; mean σ(C–C) = 0.004 Å; R factor = 0.063; wR factor = 0.154; data-to-parameter ratio = 14.1.

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).

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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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supplementary materials

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

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

P. Nithya, F. N. Khan, M. Novanna, V. R. Hathwar and S. W. Ng

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.

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).

Figures

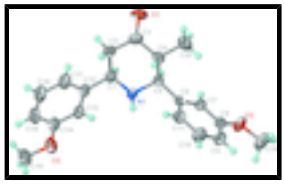


Fig. 1. Displacement ellipsoid plot (Barbour, 2001) of $C_{20}H_{23}NO_3$ at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

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

Crystal data

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

Data collection

Bruker SMART CCD area-detector	1751 reflections with $I > 2\sigma(I)$

diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 290 K

φ and ω scans

Absorption correction: none

12581 measured reflections

3148 independent reflections

R_{int} = 0.058

θ_{max} = 25.0º

θ_{min} = 1.4º

h = −33→34

k = −13→11

l = −13→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

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}

Extinction correction: none

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 (Å^2)

	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, °)

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)

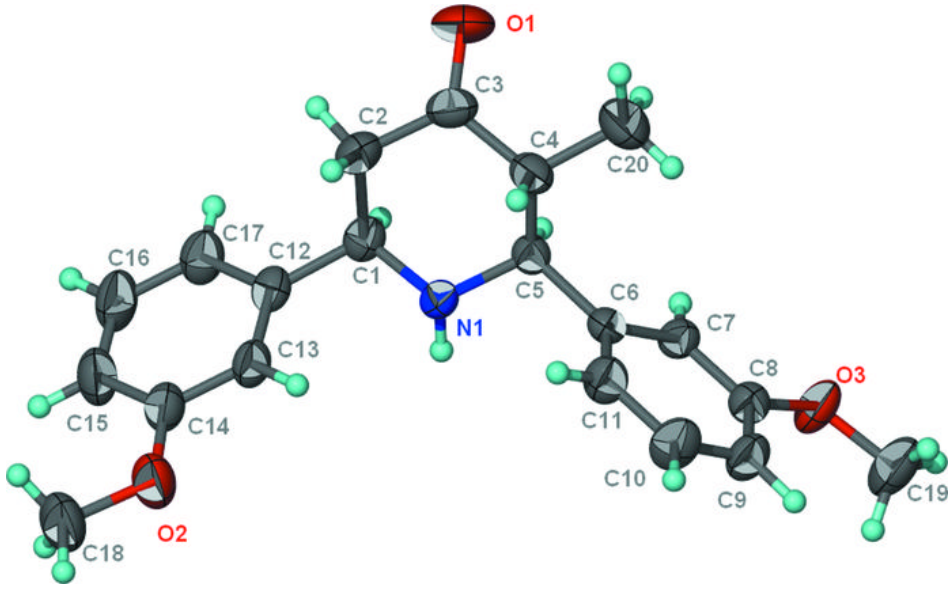
C5—C6—C7—C8	179.2 (2)	C15—C16—C17—C12	0.0 (5)
C19—O3—C8—C9	−3.2 (4)	C13—C12—C17—C16	−1.2 (4)
C19—O3—C8—C7	177.0 (3)	C1—C12—C17—C16	−178.6 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2B···O3^{i}	0.97	2.45	3.170 (4)	131
C19—H19C···O1^{ii}	0.96	2.48	3.392 (4)	159

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

Fig. 1


O1
C3
C2
C4
C20
C17
C16
C12
C1
C5
C6
C7
N1
C13
C8
O3
C15
C14
C11
C10
C9
C19
O2
C18