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2,4-Bis(3-bromophenyl)-3-azabicyclo[3.3.1]nonan-9-one

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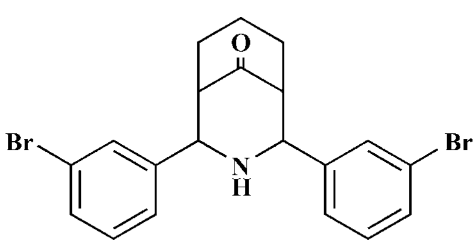
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.042; wR factor = 0.103; data-to-parameter ratio = 19.4.

The complete molecule of the title compound, $C_{20}H_{19}Br_2NO$, is generated by crystallographic mirror symmetry, with two C, one O and one N atom lying on the mirror plane. The compound exists in a twin-chair conformation with equatorial dispositions of the 3-bromophenyl groups [dihedral angle between rings = 27.37 (3)°]. The packing is stabilized by weak N—H···O and C—H···O interactions.

Related literature

For background, see: Barker *et al.* (2005); Jeyaraman & Avila (1981); Padegimas & Kovacic (1972); Smith-Verdier *et al.* (1983). For a similiar structure, see: Parthiban *et al.* (2008). For puckering parameters, see: Cremer & Pople (1975); Web & Becker (1967).



Experimental

Crystal data

$C_{20}H_{19}Br_2NO$
M_r = 449.18
Orthorhombic, *Pnma*
a = 7.1595 (6) Å
b = 24.5891 (19) Å
c = 10.2598 (6) Å
V = 1806.2 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.49 mm^{-1}
T = 298 (2) K
0.34 × 0.25 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.310, T_{max} = 0.498 (expected range = 0.277–0.445)
12758 measured reflections
2286 independent reflections
1554 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.103
S = 1.05
2286 reflections
118 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.87 (5)	2.41 (5)	3.168 (5)	145 (4)
C1—H1···O1^{ii}	0.98	2.54	3.361 (4)	142

Symmetry codes: (i) $x-1, y, z$; (ii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+\frac{5}{2}$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge the Department of Chemistry, IIT Madras, for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2838).

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supplementary materials

Acta Cryst. (2008). E**64**, o2332 [**doi:10.1107/S1600536808036660**]

2,4-Bis(3-bromophenyl)-3-azabicyclo[3.3.1]nonan-9-one

P. Parthiban, V. Ramkumar, M. S. Kim, K. T. Lim and Y. T. Jeong

Comment

Azabicyclic ketones are an important class of heterocycles due to their broad-spectrum biological activities (Jeyaraman & Avila, 1981; Barker *et al.*, 2005). Owing to the diverse possibilities in conformations, *viz*., chair-chair (Parthiban *et al.*, 2008), chair-boat (Smith-Verdier *et al.*, 1983) and boat-boat (Padegimas & Kovacic, 1972) for the azabicycle, the present crystal study was undertaken to explore the conformation, stereochemistry and bondings in the title compound, (I).

The piperidine ring in (I) adopts an ideal chair conformation with the deviation of ring atoms C3 and N1 from the C1/C2/C2^{i}/C1^{i} (i = x, 3/2-y, z) plane being 0.686 (3) and -0.702 (3) Å, respectively. The q2 and q3 values are 0.010 (3) and -0.617 (3)Å and the total puckering amplitude, Q_T = 0.617 (3)Å and θ = 180.0 (3)° (Cremer & Pople, 1975; Web & Becker, 1967).

The cyclohexane ring deviate from the ideal chair conformation by the deviation of ring atoms C3 and C5 from the C2/C4/C4^{i}/C2^{i} plane by -0.725 (4) and 0.525 (3)Å, respectively. For the cyclohexane, the q2 and q3 parameters are 0.150 (4) and 0.543 (4)Å respectively. The total puckering amplitude, Q_T = 0.563 (3)Å and θ =15.6 (4)°. Hence, the title compound, exists in a twin-chair conformation with equatorial orientations of the 3-bromophenyl groups on the heterocycle, which are orientated at an angle of 27.37 (3)° to each other. The torsion angles of C3—C2—C1—C6 and its mirror plane C3—C2^{i}—C1^{i}—C6^{i} is 174.45 (4)°. The packing is stabilized by weak N—H···O and C—H···O bonds (Table 1).

Experimental

0.1 mol of *meta* Bromobenzaldehyde and 0.05 mol of cyclohexanone were simultaneously added to a warm solution of 0.075 mol ammonium acetate in 50 ml of absolute ethanol. The mixture was gently warmed on a hot plate till the yellow colour formed during the mixing of the reactants and cooled to room temperature. Then 50 ml of ether was added and allowed to stir over night at warm condition (303–305 K). At the end, the crude azabicyclic ketone was separated by filtration and washed with 1:5 v/v ethanol-ether mixture until the solid become colourless. Colourless blocks of (I) were recrystallised from acetone.

Refinement

The nitrogen-bound H atom was located in a difference map and refined isotropically. The other hydrogen atoms were fixed geometrically (C—H = 0.93–0.98Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

Figures

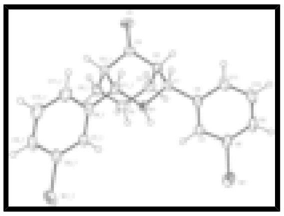

Fig. 1. The molecular structure of (I) with non-hydrogen atoms represented as 30% probability ellipsoids.

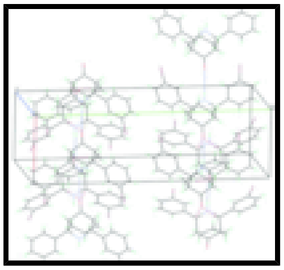

Fig. 2. Packing diagram with N—H···O (blue) and C—H···O (red) interactions.

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Crystal data

$C_{20}H_{19}Br_2NO$	F_{000} = 896
M_r = 449.18	D_x = 1.652 Mg m^{-3}
Orthorhombic, *Pnma*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2n	Cell parameters from 3647 reflections
a = 7.1595 (6) Å	θ = 3.2–23.5º
b = 24.5891 (19) Å	μ = 4.49 mm^{-1}
c = 10.2598 (6) Å	*T* = 298 (2) K
V = 1806.2 (2) Å^3	Block, colourless
Z = 4	0.34 × 0.25 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer	2286 independent reflections
Radiation source: fine-focus sealed tube	1554 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.035
T = 298(2) K	θ_{max} = 28.9º
ω scans	θ_{min} = 2.2º
Absorption correction: Multi-scan (SADABS; Bruker, 1999)	*h* = −9→9
T_{min} = 0.310, T_{max} = 0.498	*k* = −33→33
12758 measured reflections	*l* = −13→9

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.103$	$w = 1/[\sigma^2(F_o^2) + (0.0338P)^2 + 2.5194P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
2286 reflections	$\Delta\rho_{max} = 0.84$ e Å^{-3}
118 parameters	$\Delta\rho_{min} = -0.71$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
Br1	0.06375 (6)	0.580496 (16)	0.86233 (5)	0.07173 (19)
C1	0.5903 (4)	0.70119 (11)	1.1050 (3)	0.0328 (6)
H1	0.6284	0.7040	1.1965	0.039*
C2	0.7711 (4)	0.69950 (12)	1.0210 (3)	0.0353 (6)
H2	0.8451	0.6677	1.0462	0.042*
C3	0.8806 (6)	0.7500	1.0506 (4)	0.0332 (9)
C4	0.7389 (4)	0.69827 (13)	0.8727 (3)	0.0401 (7)
H4A	0.8577	0.6919	0.8298	0.048*

H4B	0.6577	0.6679	0.8521	0.048*
C5	0.6529 (6)	0.7500	0.8173 (4)	0.0418 (10)
H5A	0.6685	0.7500	0.7234	0.050*
H5B	0.5200	0.7500	0.8356	0.050*
C6	0.4822 (4)	0.64879 (11)	1.0899 (3)	0.0350 (6)
C7	0.3394 (4)	0.64162 (11)	1.0003 (3)	0.0376 (7)
H7	0.2983	0.6707	0.9498	0.045*
C8	0.2582 (4)	0.59064 (12)	0.9868 (3)	0.0431 (8)
C9	0.3157 (5)	0.54686 (12)	1.0597 (4)	0.0541 (9)
H9	0.2610	0.5129	1.0488	0.065*
C10	0.4556 (6)	0.55456 (15)	1.1489 (4)	0.0621 (11)
H10	0.4955	0.5254	1.1996	0.074*
C11	0.5382 (5)	0.60472 (14)	1.1647 (4)	0.0511 (9)
H11	0.6324	0.6091	1.2262	0.061*
N1	0.4821 (5)	0.7500	1.0736 (3)	0.0303 (7)
O1	1.0403 (4)	0.7500	1.0910 (3)	0.0493 (8)
H1A	0.376 (6)	0.7500	1.115 (4)	0.033 (12)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0813 (3)	0.0450 (2)	0.0889 (4)	−0.01841 (19)	−0.0190 (2)	−0.0091 (2)
C1	0.0371 (15)	0.0305 (14)	0.0308 (16)	0.0038 (12)	0.0021 (12)	0.0031 (12)
C2	0.0324 (15)	0.0331 (14)	0.0404 (16)	0.0064 (12)	0.0004 (12)	0.0023 (13)
C3	0.030 (2)	0.045 (2)	0.025 (2)	0.000	0.0024 (16)	0.000
C4	0.0390 (16)	0.0432 (17)	0.0382 (17)	−0.0025 (13)	0.0047 (13)	−0.0087 (14)
C5	0.039 (2)	0.058 (3)	0.028 (2)	0.000	0.0000 (19)	0.000
C6	0.0401 (16)	0.0260 (13)	0.0387 (16)	0.0037 (12)	0.0105 (13)	0.0017 (12)
C7	0.0434 (17)	0.0247 (14)	0.0447 (18)	0.0012 (12)	0.0046 (14)	0.0023 (13)
C8	0.0480 (18)	0.0284 (15)	0.0531 (19)	−0.0031 (13)	0.0108 (16)	−0.0070 (14)
C9	0.066 (2)	0.0213 (14)	0.075 (3)	−0.0032 (15)	0.016 (2)	0.0009 (16)
C10	0.071 (3)	0.0344 (18)	0.081 (3)	0.0068 (17)	−0.001 (2)	0.0204 (19)
C11	0.054 (2)	0.0394 (18)	0.060 (2)	0.0025 (15)	−0.0034 (17)	0.0154 (16)
N1	0.0286 (17)	0.0237 (16)	0.0385 (19)	0.000	0.0056 (15)	0.000
O1	0.0322 (17)	0.064 (2)	0.0520 (19)	0.000	−0.0072 (14)	0.000

Geometric parameters (Å, °)

Br1—C8	1.905 (3)	C5—H5A	0.9700
C1—N1	1.464 (3)	C5—H5B	0.9700
C1—C6	1.511 (4)	C6—C7	1.386 (4)
C1—C2	1.555 (4)	C6—C11	1.387 (4)
C1—H1	0.9800	C7—C8	1.389 (4)
C2—C3	1.500 (4)	C7—H7	0.9300
C2—C4	1.540 (4)	C8—C9	1.374 (5)
C2—H2	0.9800	C9—C10	1.369 (5)
C3—O1	1.216 (5)	C9—H9	0.9300
C3—C2^{i}	1.500 (4)	C10—C11	1.377 (5)

C4—C5	1.523 (4)	C10—H10	0.9300
C4—H4A	0.9700	C11—H11	0.9300
C4—H4B	0.9700	N1—C1^{i}	1.464 (3)
C5—C4^{i}	1.523 (4)	N1—H1A	0.87 (5)

N1—C1—C6	113.9 (2)	C4^{i}—C5—H5B	108.9
N1—C1—C2	109.9 (2)	C4—C5—H5B	108.9
C6—C1—C2	110.3 (2)	H5A—C5—H5B	107.7
N1—C1—H1	107.5	C7—C6—C11	118.8 (3)
C6—C1—H1	107.5	C7—C6—C1	123.7 (3)
C2—C1—H1	107.5	C11—C6—C1	117.4 (3)
C3—C2—C4	107.1 (3)	C6—C7—C8	119.3 (3)
C3—C2—C1	107.5 (2)	C6—C7—H7	120.3
C4—C2—C1	115.1 (2)	C8—C7—H7	120.3
C3—C2—H2	109.0	C9—C8—C7	121.8 (3)
C4—C2—H2	109.0	C9—C8—Br1	118.8 (2)
C1—C2—H2	109.0	C7—C8—Br1	119.4 (2)
O1—C3—C2	124.10 (16)	C10—C9—C8	118.3 (3)
O1—C3—C2^{i}	124.10 (17)	C10—C9—H9	120.8
C2—C3—C2^{i}	111.8 (3)	C8—C9—H9	120.8
C5—C4—C2	114.4 (3)	C9—C10—C11	121.1 (3)
C5—C4—H4A	108.7	C9—C10—H10	119.5
C2—C4—H4A	108.7	C11—C10—H10	119.4
C5—C4—H4B	108.7	C10—C11—C6	120.7 (3)
C2—C4—H4B	108.7	C10—C11—H11	119.7
H4A—C4—H4B	107.6	C6—C11—H11	119.7
C4^{i}—C5—C4	113.3 (4)	C1^{i}—N1—C1	110.1 (3)
C4^{i}—C5—H5A	108.9	C1^{i}—N1—H1A	110.7 (13)
C4—C5—H5A	108.9	C1—N1—H1A	110.7 (14)

N1—C1—C2—C3	59.2 (3)	C2—C1—C6—C11	82.1 (3)
C6—C1—C2—C3	−174.5 (2)	C11—C6—C7—C8	−0.8 (4)
N1—C1—C2—C4	−60.1 (3)	C1—C6—C7—C8	175.0 (3)
C6—C1—C2—C4	66.3 (3)	C6—C7—C8—C9	−0.1 (5)
C4—C2—C3—O1	−113.3 (4)	C6—C7—C8—Br1	−179.3 (2)
C1—C2—C3—O1	122.5 (4)	C7—C8—C9—C10	0.8 (5)
C4—C2—C3—C2^{i}	65.3 (4)	Br1—C8—C9—C10	180.0 (3)
C1—C2—C3—C2^{i}	−58.9 (4)	C8—C9—C10—C11	−0.5 (6)
C3—C2—C4—C5	−52.8 (3)	C9—C10—C11—C6	−0.4 (6)
C1—C2—C4—C5	66.7 (4)	C7—C6—C11—C10	1.1 (5)
C2—C4—C5—C4^{i}	43.3 (5)	C1—C6—C11—C10	−175.0 (3)
N1—C1—C6—C7	30.3 (4)	C6—C1—N1—C1^{i}	173.61 (18)
C2—C1—C6—C7	−93.7 (3)	C2—C1—N1—C1^{i}	−62.1 (4)
N1—C1—C6—C11	−153.8 (3)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1^{ii}	0.87 (5)	2.41 (5)	3.168 (5)	145 (4)
C1—H1···O1^{iii}	0.98	2.54	3.361 (4)	142

Symmetry codes: (ii) *x*−1, *y*, *z*; (iii) *x*−1/2, −*y*+3/2, −*z*+5/2.

Fig. 1

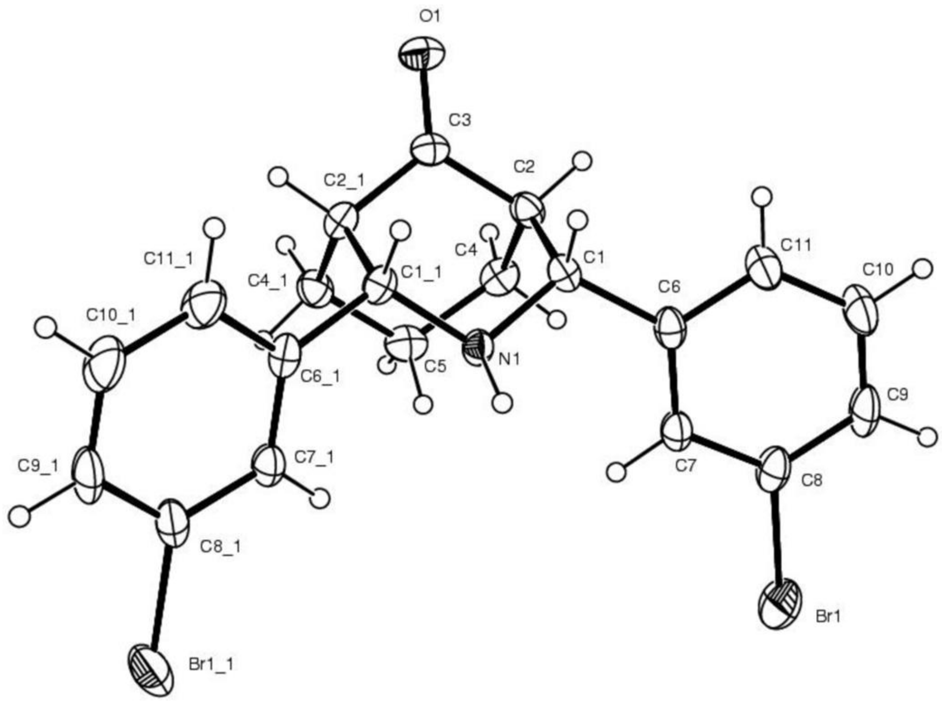
O1
C3
C2_1
C2
C11_1
C4
C1
C11
C10
C4_1
C1_1
C6
C10_1
C5
N1
C6_1
C9
C7_1
C7
C8
C9_1
C8_1
Br1
Br1_1

Fig. 2