Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,5-Dibromo-4-oxo-2,2,6,6-tetramethylpiperidin-1-yl oxide

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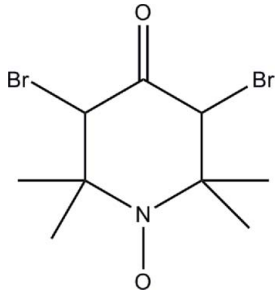
Received 12 October 2011; accepted 7 November 2011

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.034; wR factor = 0.089; data-to-parameter ratio = 14.1.

In the title compound, $C_9H_{14}Br_2NO_2$, the substituted ring exhibits a chair conformation. A crystallographic mirror plane, passing through the N atom, the O atoms and the C atom in the 4-position, bisects the molecule.

Related literature

For medical applications of similar compounds, see: Aubert *et al.* (2011); Brike (1990); Xu *et al.* (2009). For puckering parameters see: Cremer & Pople(1975).



Experimental

Crystal data

$C_9H_{14}Br_2NO_2$
M_r = 328.03
Orthorhombic, *Pnma*
a = 11.6745 (9) Å
b = 16.0848 (14) Å
c = 5.9301 (4) Å
V = 1113.57 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.26 mm^{-1}
T = 298 K
0.45 × 0.42 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.139, T_{max} = 0.355
5193 measured reflections
1018 independent reflections
774 reflections with $I > 2\sigma(I)$
R_{int} = 0.118

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.089
S = 1.04
1018 reflections
72 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2376).

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supporting information

Acta Cryst. (2011). E**67**, o3262 [https://doi.org/10.1107/S1600536811046812]

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S1. Comment

3,5-dibromo-4-oxo-2,2,6,6-tetramethylpiperidin-1-yl oxide is an important intermediate medicament. It is synthesized in a wide range of medical applications (Aubert *et al.* (2011); Brike (1990); Xu *et al.* (2009)). The complete molecule of the title compound, $C_9H_{14}Br_2NO_2$, is generated by crystallographic mirror symmetry, with two O, one C in the 3-position and one N atom lying on the mirror plane, Fig1. The substituted cyclohexyl ring adopts a chair conformation (Q_T=0.562 (4)Å, θ =19.0 (4)°, φ =180.0 (12)°), Cremer & Pople, (1975)

S2. Experimental

The title compound was synthetized by reaction between 4-Oxo-2,2,6,6-tetramethylpiperidin-1-yl oxide (2 mmol) and bromine (2 mmol), dissolved in $CH_2CH_2Cl_2$ and mixed together for 2 h. Large block crystals were precipitated, filtered ,washed with ethanol and dried in air (yield 80%).

S3. Refinement

All H atoms were positioned geometrically(C—H = 0.96–0.98 Å,) and were refined as riding, with U_{iso}(H) = 1.2U_{eq}(carrier) or 1.5U_{eq}(methyl C).

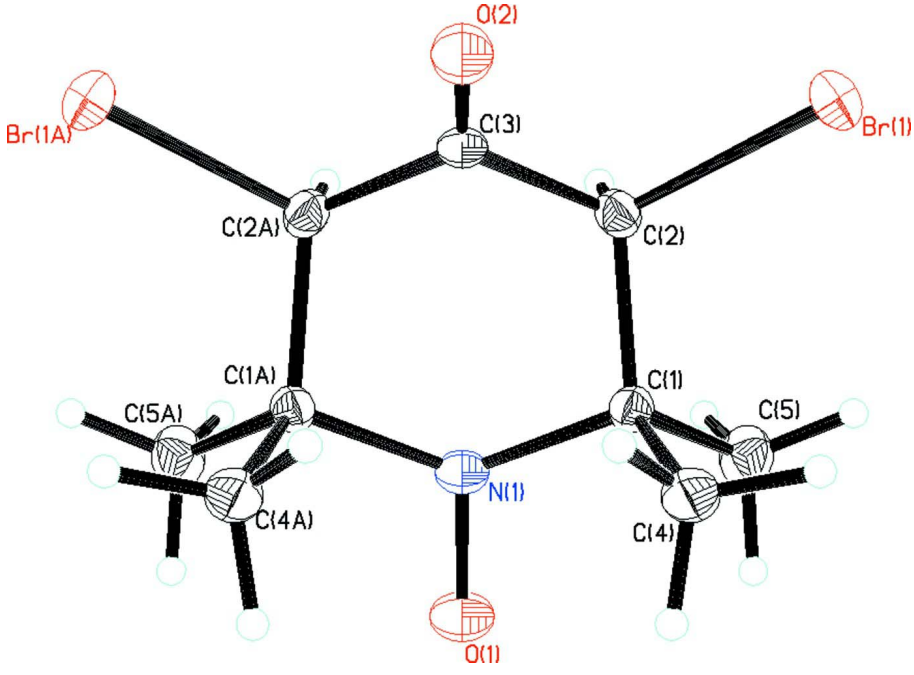

Figure 1

The molecular structure of (I) showing 30% probability displacement ellipsoids.

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Crystal data

$C_9H_{14}Br_2NO_2$
$M_r = 328.03$
Orthorhombic, *Pnma*
$a = 11.6745\ (9)$ Å
$b = 16.0848\ (14)$ Å
$c = 5.9301\ (4)$ Å
$V = 1113.57\ (15)$ Å^3
$Z = 4$
$F(000) = 644$
$D_x = 1.957$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1704 reflections
$\theta = 3.5–26.6°$
$\mu = 7.26$ mm^{-1}
$T = 298$ K
Block, orange
0.45 × 0.42 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.139$, $T_{max} = 0.355$
5193 measured reflections
1018 independent reflections
774 reflections with $I > 2\sigma(I)$
$R_{int} = 0.118$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.5°$
$h = -13\rightarrow12$
$k = -19\rightarrow18$
$l = -6\rightarrow7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.089$
$S = 1.04$
1018 reflections
72 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
$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.64$ 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}
Br1	0.28737 (4)	0.42632 (3)	0.59566 (7)	0.0414 (2)
N1	0.4892 (4)	0.2500	0.2492 (7)	0.0268 (11)

O1	0.5770 (4)	0.2500	0.1217 (6)	0.0448 (11)
O2	0.2994 (3)	0.2500	0.7814 (7)	0.0384 (10)
C1	0.4591 (3)	0.3333 (2)	0.3498 (6)	0.0237 (9)
C2	0.3343 (3)	0.3265 (2)	0.4364 (6)	0.0263 (9)
H2	0.2844	0.3206	0.3045	0.032*
C3	0.3177 (4)	0.2500	0.5827 (10)	0.0277 (13)
C4	0.5446 (3)	0.3542 (3)	0.5367 (7)	0.0354 (10)
H4A	0.5310	0.3188	0.6643	0.053*
H4B	0.5354	0.4112	0.5808	0.053*
H4C	0.6212	0.3456	0.4824	0.053*
C5	0.4652 (4)	0.3975 (3)	0.1615 (8)	0.0418 (11)
H5A	0.5425	0.4012	0.1069	0.063*
H5B	0.4416	0.4507	0.2182	0.063*
H5C	0.4155	0.3811	0.0405	0.063*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0384 (3)	0.0281 (3)	0.0576 (4)	0.00706 (19)	0.00867 (18)	−0.0047 (2)
N1	0.024 (2)	0.033 (3)	0.023 (3)	0.000	0.0032 (17)	0.000
O1	0.039 (3)	0.050 (3)	0.046 (3)	0.000	0.0234 (19)	0.000
O2	0.048 (3)	0.034 (2)	0.033 (3)	0.000	0.0153 (19)	0.000
C1	0.025 (2)	0.022 (2)	0.024 (2)	−0.0017 (17)	0.0009 (14)	−0.0011 (16)
C2	0.023 (2)	0.024 (2)	0.033 (2)	0.0032 (17)	−0.0028 (14)	−0.0031 (17)
C3	0.011 (3)	0.026 (3)	0.046 (4)	0.000	−0.001 (2)	0.000
C4	0.024 (2)	0.034 (2)	0.047 (3)	−0.001 (2)	−0.0046 (17)	−0.007 (2)
C5	0.053 (3)	0.034 (2)	0.039 (3)	−0.001 (2)	0.006 (2)	0.007 (2)

Geometric parameters (Å, º)

Br1—C2	1.942 (4)	C2—H2	0.9800
N1—O1	1.274 (5)	C3—C2^{i}	1.518 (5)
N1—C1^{i}	1.509 (4)	C4—H4A	0.9600
N1—C1	1.509 (4)	C4—H4B	0.9600
O2—C3	1.198 (6)	C4—H4C	0.9600
C1—C5	1.523 (6)	C5—H5A	0.9600
C1—C4	1.529 (5)	C5—H5B	0.9600
C1—C2	1.548 (5)	C5—H5C	0.9600
C2—C3	1.518 (5)		
O1—N1—C1^{i}	115.0 (2)	O2—C3—C2^{i}	125.8 (2)
O1—N1—C1	115.0 (2)	O2—C3—C2	125.8 (2)
C1^{i}—N1—C1	125.3 (4)	C2^{i}—C3—C2	108.3 (5)
N1—C1—C5	107.5 (3)	C1—C4—H4A	109.5
N1—C1—C4	109.2 (3)	C1—C4—H4B	109.5
C5—C1—C4	110.6 (3)	H4A—C4—H4B	109.5
N1—C1—C2	106.7 (3)	C1—C4—H4C	109.5
C5—C1—C2	109.6 (3)	H4A—C4—H4C	109.5

C4—C1—C2	112.9 (3)	H4B—C4—H4C	109.5
C3—C2—C1	111.5 (3)	C1—C5—H5A	109.5
C3—C2—Br1	110.9 (3)	C1—C5—H5B	109.5
C1—C2—Br1	111.6 (3)	H5A—C5—H5B	109.5
C3—C2—H2	107.5	C1—C5—H5C	109.5
C1—C2—H2	107.5	H5A—C5—H5C	109.5
Br1—C2—H2	107.5	H5B—C5—H5C	109.5
O1—N1—C1—C5	46.0 (5)	C4—C1—C2—C3	−69.8 (4)
C1^{i}—N1—C1—C5	−159.6 (3)	N1—C1—C2—Br1	174.9 (2)
O1—N1—C1—C4	−74.1 (4)	C5—C1—C2—Br1	−69.0 (3)
C1^{i}—N1—C1—C4	80.3 (5)	C4—C1—C2—Br1	54.8 (4)
O1—N1—C1—C2	163.5 (4)	C1—C2—C3—O2	111.8 (5)
C1^{i}—N1—C1—C2	−42.1 (6)	Br1—C2—C3—O2	−13.2 (6)
N1—C1—C2—C3	50.2 (4)	C1—C2—C3—C2^{i}	−65.4 (5)
C5—C1—C2—C3	166.4 (3)	Br1—C2—C3—C2^{i}	169.5 (2)

Symmetry code: (i) x, $-y+1/2$, z.