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3,5-Dibromo-2,2,6,6-tetramethylpiperidin-4-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.043
wR factor = 0.090
Data-to-parameter ratio = 17.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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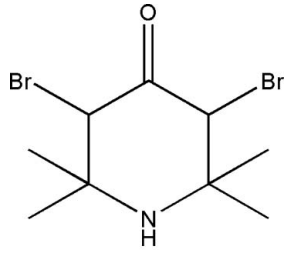
In the title compound, $C_9H_{15}Br_2NO$, the piperidin-4-one ring adopts a chair conformation.

Comment

2,2,6,6-Tetramethylpiperidin-4-one, (I), plays an important role as a starting material in the research and development of HALS (hindered amine light stabilizers) (Yoshihiko *et al.*, 2004). In our studies on HALS, many derivatives of (I) have been prepared. The title compound, (II) (Fig. 1), is one of these compounds and its structure is reported here.



(II)

In the molecule of (II), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The piperidin-4-one ring adpots a chair conformation. The N1—H1*a* group participates in a very weak intermolecular hydrogen bond with atom O1 of an adjacent molecule (Table 1); this is possible because of its axial location with respect to the six-membered ring.

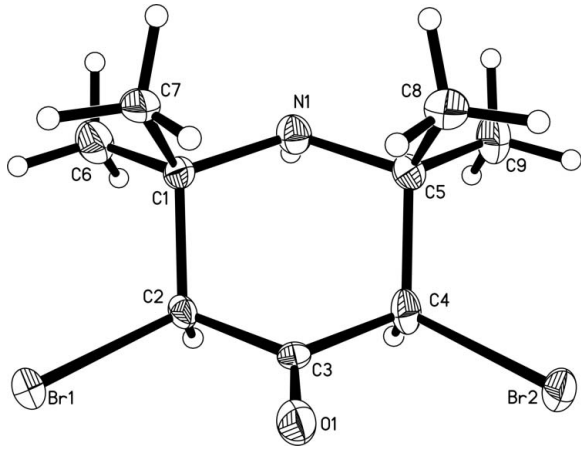


Figure 1
The molecular structure of (II), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

Experimental

The title compound was synthesized by the reaction of 2,2,6,6-tetramethylpiperidin-4-one with bromine in the presence of acetic acid (Couet *et al.*, 1985). Colourless prisms of (II) were obtained by recrystallizion from an acetone solution (m.p. 418 K). Analysis calculated for $C_9H_{15}Br_2NO$: C 34.53, H 4.83, Br 51.05, N 4.47%; found: C 34.79, H 4.56, N 4.38%.

Crystal data

$C_9H_{15}Br_2NO$
M_r = 313.04
Monoclinic, $P2_1/c$
a = 8.8677 (18) Å
b = 21.968 (4) Å
c = 6.0078 (12) Å
β = 107.78 (3)°
V = 1114.5 (4) Å^3
Z = 4
D_x = 1.866 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 7.24 mm^{-1}
T = 293 (2) K
Prism, colourless
0.34 × 0.18 × 0.16 mm

Data collection

Rigaku Saturn diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.210, T_{max} = 0.314
8984 measured reflections
2181 independent reflections
1667 reflections with $I > 2\sigma(I)$
R_{int} = 0.079
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.090
S = 0.99
2181 reflections
125 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.86 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}	0.86 (4)	2.64 (4)	3.488 (6)	169 (6)

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

The N-bound H atom was located in a difference map and its position was refined freely with U_{iso}(H) = 1.5U_{eq}(N). Other H atoms were positioned geometrically (C—H = 0.96–0.98 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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