Crystal structure of *rel*-(2*R*,3a*S*)-2-bromomethyl-3,3a,4,5,6,7-hexahydro-2*H*-indole 1-oxide, $C_9H_{14}BrNO$

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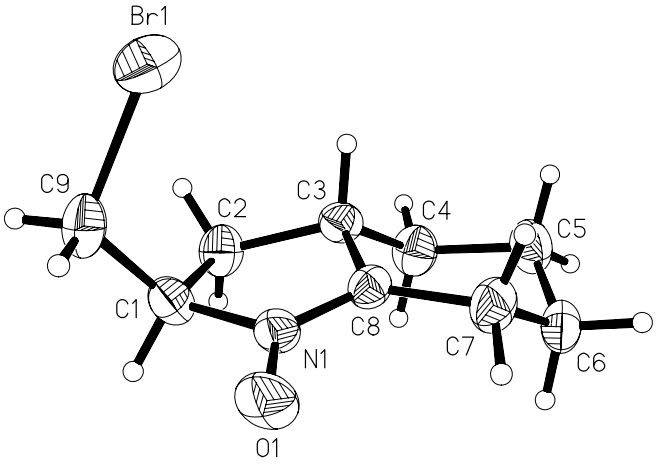

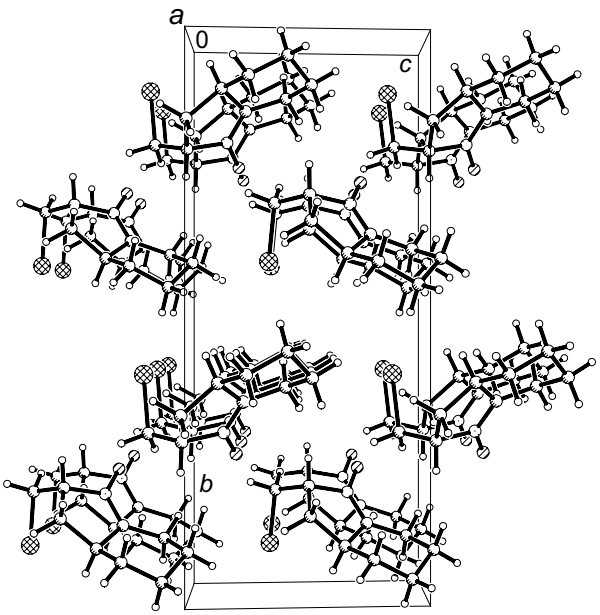

Abstract

$C_9H_{14}BrNO$, monoclinic, *C*1*c*1 (no. 9), a = 6.079(1) Å, b = 19.095(4) Å, c = 8.169(1) Å, β = 95.23(1)°, V = 944.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.047, $wR_{ref}(F^2)$ = 0.120, T = 293 K.

Source of material

The title compound has been obtained by bromocyclization of 2-allylcyclohexanone oxime [1-4]. The two diastereoisomers (ratio 67:33) were separated by column chromatography using ethyl acetate/methanol (7:3), whereby crystallization of the minor isomer from dichloromethane furnished the title nitrone in the form of brownish crystals (m.p. 375-376 K).

Discussion

The six-membered ring system shows a chair conformation and the pyrrolidine ring has an envelope conformation, where C2 is out of plane. The double bond N1=C8 is clearly characterized by the distance of 1.298(7) Å. The N—O distance of the neighboring *N*-oxide function is 1.309 (6) Å. In the packing diagram of the cell plot we observe an antiparallel orientation of the molecules along the *c* axis and a face-to-face stacking of the molecules along the *a* axis.

Table 1. Data collection and handling.

Crystal:	brownish plate, size 0.15 × 0.6 × 1.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	43.06 cm^{-1}
Diffractometer, scan mode:	Nicolet P3, Wyckoff
$2\theta_{max}$:	59.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1502, 1502
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1255
$N(param)_{refined}$:	110
Programs:	SHELXS-97 [5], SHELXL-97 [6], SHELXTL-plus [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	0.3886	0.2451	0.5011	0.040
H(2A)	4*a*	0.5537	0.3652	0.4043	0.044
H(2B)	4*a*	0.6804	0.3132	0.5301	0.044
H(3)	4*a*	0.4631	0.4377	0.6016	0.035
H(4A)	4*a*	0.8271	0.4384	0.7033	0.046
H(4B)	4*a*	0.8063	0.3654	0.7890	0.046
H(5A)	4*a*	0.8072	0.4549	0.9871	0.051
H(5B)	4*a*	0.6023	0.4902	0.8893	0.051
H(6A)	4*a*	0.4855	0.4264	1.1105	0.046
H(6B)	4*a*	0.5920	0.3580	1.0463	0.046
H(7A)	4*a*	0.2230	0.3483	0.9625	0.046
H(7B)	4*a*	0.2097	0.4260	0.8988	0.046
H(9A)	4*a*	0.0370	0.2768	0.3822	0.049
H(9B)	4*a*	0.2194	0.2900	0.2620	0.049

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	4*a*	0.07072(3)	0.40354(3)	0.34010(3)	0.0468(4)	0.0582(4)	0.0475(3)	0.0068(4)	0.0028(2)	0.0103(3)
N(1)	4*a*	0.2530(8)	0.3079(2)	0.6646(5)	0.035(2)	0.026(2)	0.029(2)	–0.003(2)	0.008(2)	0.001(2)
O(1)	4*a*	0.0931(8)	0.2684(2)	0.7097(6)	0.047(3)	0.035(2)	0.052(2)	–0.016(2)	0.014(2)	0.002(2)
C(1)	4*a*	0.3411(9)	0.2941(3)	0.5032(6)	0.036(3)	0.027(2)	0.037(2)	–0.002(2)	0.009(2)	–0.004(2)
C(2)	4*a*	0.547(1)	0.3409(3)	0.5080(6)	0.035(3)	0.043(3)	0.032(2)	0.001(3)	0.011(2)	–0.005(2)
C(3)	4*a*	0.521(1)	0.3935(3)	0.6489(6)	0.031(3)	0.027(2)	0.030(2)	0.000(2)	0.009(2)	0.003(2)
C(4)	4*a*	0.730(1)	0.4089(3)	0.7607(7)	0.035(3)	0.044(3)	0.037(3)	–0.006(3)	0.003(2)	–0.003(2)
C(5)	4*a*	0.672(1)	0.4456(3)	0.9177(7)	0.047(4)	0.040(3)	0.038(3)	–0.008(3)	0.002(2)	–0.010(2)
C(6)	4*a*	0.518(2)	0.4014(3)	1.0123(7)	0.048(4)	0.039(3)	0.028(2)	–0.002(3)	0.005(3)	–0.008(2)
C(7)	4*a*	0.302(1)	0.3845(3)	0.9087(6)	0.042(3)	0.043(3)	0.030(2)	–0.001(3)	0.013(2)	–0.002(2)
C(8)	4*a*	0.3492(9)	0.3603(3)	0.7429(5)	0.027(2)	0.030(2)	0.025(2)	0.001(2)	0.004(2)	0.001(2)
C(9)	4*a*	0.164(1)	0.3054(3)	0.3637(7)	0.044(3)	0.051(3)	0.030(2)	–0.004(3)	0.007(2)	–0.011(2)

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