Z. Kristallogr. NCS 221 (2006) 217-218 / DOI 10.1524/ncrs.2006.0045

Crystal structure of *rel*-(2*R*,3*S*)-2-bromomethyl-3-phenyl-3,4-dihydro-2*H*-pyrrole 1-oxide, $C_{11}H_{12}BrNO$

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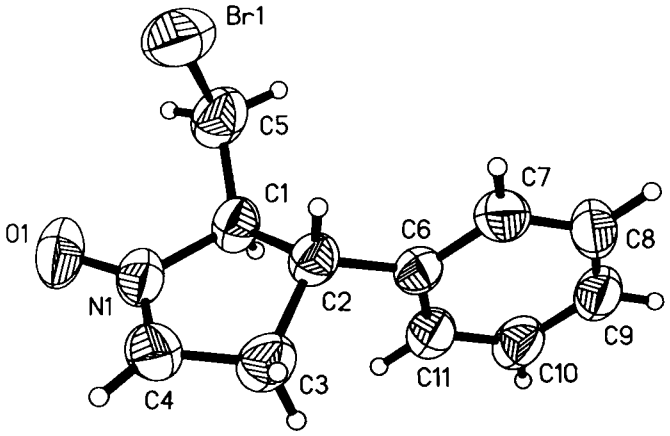
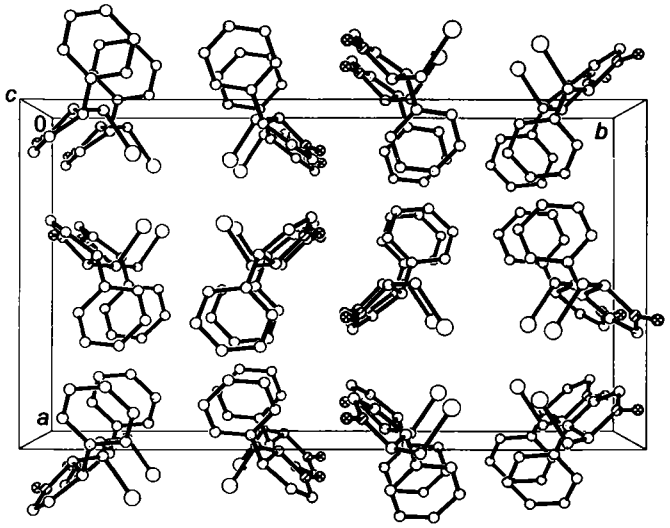
Received April 28, 2006, accepted and available on-line May 15, 2006; CCDC no. 1267/1776





Abstract

$C_{11}H_{12}BrNO$, orthorhombic, *Pccn* (no. 56), a = 13.1274(7) Å, b = 23.309(1) Å, c = 6.995(1) Å, V = 2140.4 Å^3, Z = 8, $R_{gt}(F)$ = 0.065, $wR_{ref}(F^2)$ = 0.207, T = 293 K.

Source of material

The title compound has been obtained by bromocyclization of 3-phenyl-4-pentenal oxime [1-7]. The two diastereoisomers (ratio 40:60) were separated by column chromatography (ethyl acetate/methanol), whereby crystallization of the major isomer from dichloromethane furnished the title nitrone in the form of colorless crystals (m.p. 398-401 K).

Discussion

The pyrroline ring system has an envelope conformation, where C2 is situated out-of-plane. Both ring systems of the molecule have a nearly perfect perpendicular orientation of 88.9(2)° (figure, top). The double bond N1=C4 is clearly characterized by a distance of 1.287(3) Å. The N—O distance of the neighboring *N*-oxide function is 1.293(5) Å. In the *a,b* view of the cell plot we observe alternate polar and non-polar channels along the *c* axis. The non-polar channels are built up by the phenyl moieties and the polar ones by the bromomethyl-nitrone fragments. There are also polar layers in the *a,c* plane along the *b* axis established by the *N*-oxide functions (figure, bottom).

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.15 × 0.20 × 0.50 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	49.61 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	135.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2505, 1875
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1449
$N(param)_{refined}$:	128
Programs:	SHELXS-97 [8], SHELXL-97 [9], SHELXTL-Plus [10]

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

Atom	Site	x	y	z	U_{iso}
H(1)	8*e*	0.4645	0.5698	−0.0710	0.061
H(2)	8*e*	0.5986	0.6478	−0.2383	0.057
H(3A)	8*e*	0.5829	0.5429	−0.4369	0.076
H(3B)	8*e*	0.6791	0.5833	−0.4208	0.076
H(4)	8*e*	0.7114	0.5062	−0.1937	0.080
H(5A)	8*e*	0.5122	0.6086	0.2193	0.084
H(5B)	8*e*	0.4681	0.6561	0.0843	0.084
H(7)	8*e*	0.5013	0.7150	−0.4017	0.068
H(8)	8*e*	0.3632	0.7412	−0.5879	0.080
H(9)	8*e*	0.2439	0.6742	−0.6765	0.077
H(10)	8*e*	0.2581	0.5808	−0.5707	0.074
H(11)	8*e*	0.3953	0.5538	−0.3849	0.063

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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)	8*e*	0.64818(6)	0.67571(3)	0.1394(1)	0.0918(6)	0.0904(6)	0.0910(7)	0.0062(3)	−0.0227(4)	−0.0335(4)
N(1)	8*e*	0.6112(3)	0.5451(2)	−0.0317(6)	0.051(2)	0.059(2)	0.054(3)	0.001(2)	−0.012(2)	0.006(2)
O(1)	8*e*	0.6199(3)	0.5214(2)	0.1346(5)	0.079(2)	0.079(2)	0.071(3)	−0.003(2)	−0.024(2)	0.022(2)
C(1)	8*e*	0.5302(3)	0.5895(2)	−0.0617(6)	0.045(2)	0.065(2)	0.042(3)	0.005(2)	0.000(2)	0.006(2)
C(2)	8*e*	0.5559(3)	0.6138(2)	−0.2572(6)	0.046(2)	0.057(2)	0.040(2)	0.003(2)	0.001(2)	0.001(2)
C(3)	8*e*	0.6227(4)	0.5666(3)	−0.3507(7)	0.055(2)	0.087(4)	0.048(3)	0.014(2)	0.001(2)	−0.008(2)
C(4)	8*e*	0.6592(4)	0.5331(2)	−0.1864(9)	0.056(3)	0.070(3)	0.075(4)	0.017(2)	−0.012(2)	−0.013(3)
C(5)	8*e*	0.5251(4)	0.6303(3)	0.1035(8)	0.072(3)	0.093(4)	0.045(3)	0.014(3)	0.002(2)	−0.003(3)
C(6)	8*e*	0.4644(3)	0.6316(2)	−0.3744(6)	0.052(2)	0.051(2)	0.035(2)	0.005(2)	0.003(2)	−0.000(2)
C(7)	8*e*	0.4531(4)	0.6876(2)	−0.4357(7)	0.069(3)	0.050(2)	0.050(3)	0.000(2)	0.003(2)	−0.002(2)
C(8)	8*e*	0.3701(4)	0.7034(2)	−0.5478(8)	0.087(4)	0.064(3)	0.050(3)	0.027(3)	0.003(3)	0.011(2)
C(9)	8*e*	0.2985(4)	0.6635(2)	−0.5996(7)	0.067(3)	0.080(3)	0.045(3)	0.024(3)	−0.008(2)	−0.001(2)
C(10)	8*e*	0.3074(4)	0.6077(2)	−0.5376(7)	0.055(2)	0.077(3)	0.052(3)	0.004(2)	−0.012(2)	−0.005(2)
C(11)	8*e*	0.3897(4)	0.5916(2)	−0.4260(7)	0.052(2)	0.059(2)	0.046(3)	0.001(2)	−0.004(2)	0.001(2)

Acknowledgments. For financial support of this work we are grateful to Fonds der Chemischen Industrie, to Bayer AG (Wuppertal), and to the EU COST D13 action (Molecules for Health).

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