Crystal structure of rel-(2R,3S)-2-bromomethyl-3-phenyl-3,4-dihydro-2H-pyrrole 1-oxide, C₁₁H₁₂BrNO

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Abstract
C₁₁H₁₂BrNO, orthorhombic, Pccn (no. 56), a = 13.1274(7) Å, b = 23.309(1) Å, c = 6.995(1) Å, V = 2140.4 Å³, Z = 8, Rₑ(F) = 0.065, wRₑ(F²) = 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 nitrene 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 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 x 0.20 x 0.50 mm |
| Wavelength:              | Cu Kα radiation (1.54178 Å) |
| μ:                       | 49.61 cm⁻¹ |
| Diffractometer, scan mode: | Siemens P4, ω |
| 2θmax:                   | 135.96°* |
| N(hkl)measured, N(hkl)unique: | 2505, 1875 |
| Criterion for I₁₀₀, N(hkl)refined: | I₁₀₀ > 2 σ(I₁₀₀), 1449 |
| Programs:                | SHELXS-97 [8], SHELXL-97 [9], SHELXTL-Plus [10] |

Table 2. Atomic coordinates and displacement parameters (in Å²).

<table>
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<tr>
<th>Atom</th>
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<th>y</th>
<th>z</th>
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* Correspondence author (e-mail: jager.ioc@po.uni-stuttgart.de)
### Table 3. Atomic coordinates and displacement parameters (in Å²).

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### Acknowledgments.
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### References