Crystal structure of *rel*-(3*aS*,6*aS*)-3-(4-methoxyphenyl)-4,6*a*-dihydro-3*aH*-cyclopenta[*d*]isoxazole, C₁₃H₁₃NO₂

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Received December 17, 2004, accepted and available on-line May 2, 2005; CCDC no. 1267/1448

Abstract

C₁₃H₁₃NO₂, monoclinic, *P*12₁/*c*1 (no. 14), *a* = 9.823(3) Å, *b* = 4.379(1) Å, *c* = 25.698(7) Å, β = 96.51(2)°, *V* = 1098.2 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.070, *wR*_{ref}(*F*²) = 0.176, *T* = 293 K.

Source of material

The title compound was obtained by 1,3-dipolar nitrile oxide cycloaddition of 4-methoxybenzonitrile oxide released from 4-methoxy-benzhydroxamic acid chloride [1–5] through slow addition of triethylamine solution in chloroform [6–7] and freshly distilled cyclopentadiene (excess). Purification by column chromatography and crystallization from ethyl acetate and petroleum ether furnished the title dihydro-cyclopentaisoxazole derivative in the form of colorless crystals (m.p. 348–349 K) [8].

Discussion

The anisyl groups show a coplanar orientation to the isoxazoline moieties (figure, top). In the packing diagram of the cell plot we observe layers built up by the phenyl groups in the *a,b* plane stacking along the *c* axis (figure, bottom). We found also a bi-layer-type orientation of the bicyclic systems in the *a,b* plane.

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.10 × 0.20 × 0.75 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ :	0.88 cm ^{−1}
Diffractometer, scan mode:	Nicolet P3, Wyckoff
2 θ _{max} :	46°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	1624, 1522
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 959
<i>N</i> (<i>param</i>) _{refined} :	146
Programs:	SHELXS-97 [9], SHELXL-97 [10]

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(2)	4e	0.2432	0.4872	0.1122	0.073
H(3A)	4e	0.4082	0.9877	0.1253	0.087
H(3B)	4e	0.4469	0.6845	0.0970	0.087
H(4)	4e	0.4202	1.1108	0.0319	0.096
H(5)	4e	0.1978	1.0443	−0.0095	0.094
H(6)	4e	0.1129	0.5904	0.0370	0.087
H(8)	4e	0.0604	1.1638	0.2216	0.075
H(9)	4e	0.1239	1.1708	0.3102	0.073
H(11)	4e	0.4362	0.6136	0.2901	0.076
H(12)	4e	0.3707	0.6049	0.2018	0.071
H(13A)	4e	0.2968	0.9982	0.4319	0.126
H(13B)	4e	0.1626	0.9455	0.3938	0.126
H(13C)	4e	0.2504	1.2407	0.3886	0.126

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(1)	4e	0.1629(3)	0.872(1)	0.1450(2)	0.039(2)	0.055(3)	0.073(3)	−0.001(2)	0.010(2)	0.008(2)
O(1)	4e	0.0310(3)	0.9529(9)	0.0710(1)	0.054(2)	0.138(3)	0.073(2)	0.021(2)	0.001(1)	0.014(2)
N(1)	4e	0.0533(3)	1.004(1)	0.1257(1)	0.050(2)	0.100(3)	0.069(2)	0.017(2)	0.012(2)	0.011(2)
O(2)	4e	0.3315(3)	0.8715(8)	0.3611(1)	0.063(2)	0.095(3)	0.069(2)	0.002(2)	0.006(2)	−0.002(2)
C(2)	4e	0.2375(4)	0.707(1)	0.1053(2)	0.057(2)	0.052(3)	0.072(3)	0.008(2)	0.001(2)	−0.003(2)
C(3)	4e	0.3783(4)	0.844(1)	0.0976(2)	0.047(2)	0.098(4)	0.072(3)	0.008(2)	0.011(2)	−0.011(3)
C(4)	4e	0.3534(5)	0.998(1)	0.0463(2)	0.072(3)	0.106(4)	0.068(3)	0.001(3)	0.029(2)	−0.011(3)

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Table 3. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(5)	4e	0.2292(5)	0.963(1)	0.0231(2)	0.077(3)	0.104(4)	0.055(2)	0.017(3)	0.011(2)	-0.003(3)
C(6)	4e	0.1448(4)	0.778(1)	0.0553(2)	0.062(3)	0.073(3)	0.079(3)	0.008(3)	-0.006(2)	-0.013(3)
C(7)	4e	0.2074(3)	0.8846(9)	0.2016(2)	0.040(2)	0.052(3)	0.066(2)	-0.006(2)	0.008(2)	0.009(2)
C(8)	4e	0.1363(4)	1.051(1)	0.2353(2)	0.041(2)	0.067(3)	0.080(3)	0.006(2)	0.014(2)	0.006(3)
C(9)	4e	0.1736(4)	1.056(1)	0.2885(2)	0.049(2)	0.062(3)	0.073(3)	-0.002(2)	0.015(2)	-0.004(2)
C(10)	4e	0.2852(4)	0.888(1)	0.3093(2)	0.045(2)	0.060(3)	0.062(3)	-0.007(2)	0.008(2)	0.005(2)
C(11)	4e	0.3594(4)	0.723(1)	0.2763(2)	0.052(2)	0.067(3)	0.071(3)	0.005(2)	0.002(2)	0.003(2)
C(12)	4e	0.3206(4)	0.720(1)	0.2234(2)	0.049(2)	0.059(3)	0.071(3)	0.005(2)	0.009(2)	0.001(2)
C(13)	4e	0.2540(5)	1.027(1)	0.3968(2)	0.079(3)	0.108(4)	0.069(3)	0.000(3)	0.018(2)	-0.008(3)

Acknowledgments. We are grateful to Fonds der Chemischen Industrie and to Bayer AG for financial support.

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