Crystal structure of (3*R*,1'*S*)-3-(1',2'-*O*-cyclohexylidenedioxyethyl)-2,5,5-trimethyl-3-nitromethyltetrahydro-1,2-oxazole, C₁₅H₂₆N₂O₅

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Abstract

C₁₅H₂₆N₂O₅, orthorhombic, *P*2₁2₁2₁ (no. 19), *a* = 8.070(2) Å, *b* = 9.942(3) Å, *c* = 21.151(4) Å, *V* = 1696.9 Å³, *Z* = 4, *R*_g(*F*) = 0.069, *wR*_{ref}(*F*²) = 0.142, *T* = 293 K.

Source of material

The title compound has been obtained by nucleophilic addition of methane nitronate (CH₃NO₂/base) to the *N*-methylisoxazolinium salt formed by *N*-methylation of (1'*S*)-3-(1',2'-*O*-cyclohexylidenedioxyethyl)-5,5-dimethyl-4,5-dihydroisoxazole with Meerwein's salt Me₃OBf₄ [1–5]. Separation of the two diastereoisomers and purification by MPLC and crystallization from petroleum ether gave the title nitromethylisoxazolidine compound in the form of colourless crystals [1] (m.p. 320–322 K); [*α*]_D²⁰ = –56.0 (*c* = 1.45, CHCl₃).

Experimental details

H atoms were located on difference fourier map, but refined with fixed individual displacement parameters using a riding model with a *d*(C–H) ranging from 0.96 to 0.98 Å.

Discussion

The title compound crystallizes with one molecule in the asymmetric unit. The isoxazolidine ring shows an envelope conformation, where N1 is 0.68(3) Å out-of-plane. The dioxolane moiety also has an envelope conformation, where O2 is 0.46(4) Å out-of-plane. The best planes of both ring systems have a nearly perpendicular orientation to each other indicated by a dihedral angle of 83.9(1)°. The crystal structure is also stabilized by two weak hydrogen bond contacts, where the methylene groups of the cyclohexylidene moiety act as a donors. The acceptors are the oxygen atoms O1 of the isoxazolidine and O4 of the nitro group. The

H11A...O1 distance is 2.71 Å and the angle C11–H11A...O1 is 168°. The H14B...O4 distance is 2.75 Å and the angle C14–H14B...O4 is 156°. The strong vibrational behaviour of the nitro group is indicated by large displacement parameters of its oxygen atoms.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.4 × 0.6 × 0.7 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
<i>μ</i> :	0.92 cm ^{−1}
Diffractometer, scan mode:	Nicolet P3, Wyckoff
2 θ _{max} :	58°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	5042, 4514
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 2867
<i>N</i> (<i>param</i>) _{refined} :	200
Programs:	SHELXS-97 [6], SHELXL-97 [7], SHELXTL [8]

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(2A)	4a	−0.0243	0.7880	0.7947	0.057
H(2B)	4a	−0.2006	0.7221	0.7857	0.057
H(4A)	4a	−0.0756	0.7400	1.0178	0.103
H(4B)	4a	0.0007	0.6063	0.9914	0.103
H(4C)	4a	−0.1913	0.6294	0.9887	0.103
H(5A)	4a	−0.4427	0.8234	0.8354	0.140
H(5B)	4a	−0.3788	0.9422	0.7932	0.140
H(5C)	4a	−0.4253	0.9680	0.8642	0.140
H(6A)	4a	0.0168	0.9725	0.8674	0.136
H(6B)	4a	−0.1374	1.0614	0.8842	0.136
H(6C)	4a	−0.0908	1.0357	0.8133	0.136
H(7A)	4a	−0.1949	0.4893	0.9108	0.060
H(7B)	4a	−0.3222	0.5881	0.8795	0.060
H(8)	4a	0.0771	0.5380	0.8101	0.053
H(9A)	4a	0.2716	0.6966	0.8110	0.070
H(9B)	4a	0.2250	0.7463	0.8795	0.070
H(11A)	4a	0.3156	0.4890	0.9921	0.072
H(11B)	4a	0.4711	0.4173	0.9628	0.072
H(12A)	4a	0.3259	0.2601	1.0229	0.087
H(12B)	4a	0.1616	0.2917	0.9862	0.087
H(13A)	4a	0.2674	0.0925	0.9475	0.091
H(13B)	4a	0.4400	0.1599	0.9345	0.091
H(14A)	4a	0.1442	0.2182	0.8682	0.086
H(14B)	4a	0.2995	0.1460	0.8392	0.086
H(15A)	4a	0.4544	0.3432	0.8456	0.072
H(15B)	4a	0.2905	0.3753	0.8086	0.072

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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
O(1)	4a	−0.2134(3)	0.8199(2)	0.91747(9)	0.068(1)	0.053(1)	0.046(1)	0.010(1)	0.007(1)	0.0023(9)
N(1)	4a	−0.0676(3)	0.7343(2)	0.9233(1)	0.055(1)	0.051(1)	0.042(1)	0.001(1)	−0.001(1)	0.001(1)
C(1)	4a	−0.2021(4)	0.8733(3)	0.8543(1)	0.068(2)	0.046(2)	0.047(2)	0.004(2)	0.003(2)	0.004(1)
O(2)	4a	0.1188(2)	0.4812(2)	0.89831(9)	0.0341(9)	0.050(1)	0.058(1)	−0.0019(8)	0.0040(8)	0.0110(9)
C(2)	4a	−0.1231(4)	0.7575(3)	0.8166(1)	0.052(2)	0.047(1)	0.043(1)	0.001(1)	−0.002(1)	0.003(1)
N(2)	4a	−0.2296(3)	0.4515(3)	0.8171(2)	0.055(2)	0.071(2)	0.098(2)	−0.026(2)	−0.002(2)	−0.009(2)
C(3)	4a	−0.0792(3)	0.6495(3)	0.8663(1)	0.037(1)	0.047(1)	0.039(1)	−0.006(1)	0.001(1)	0.002(1)
O(3)	4a	0.3639(2)	0.5834(2)	0.8788(1)	0.038(1)	0.062(1)	0.118(2)	−0.010(1)	−0.010(1)	0.016(1)
O(4)	4a	−0.2865(6)	0.4957(4)	0.7701(2)	0.237(5)	0.125(3)	0.080(2)	−0.098(3)	−0.032(3)	0.005(2)
C(4)	4a	−0.0850(5)	0.6721(3)	0.9857(1)	0.093(3)	0.074(2)	0.040(2)	0.016(2)	0.001(2)	0.006(2)
C(5)	4a	−0.3783(5)	0.9046(5)	0.8350(2)	0.090(3)	0.115(3)	0.075(2)	0.049(3)	0.001(2)	0.003(2)
O(5)	4a	−0.1891(5)	0.3365(3)	0.8232(2)	0.148(3)	0.082(2)	0.230(4)	0.024(2)	−0.065(3)	−0.065(3)
C(6)	4a	−0.0935(6)	0.9970(3)	0.8549(2)	0.147(4)	0.051(2)	0.074(2)	−0.024(2)	0.011(3)	0.002(2)
C(7)	4a	−0.2168(3)	0.5429(3)	0.8734(1)	0.040(1)	0.054(2)	0.057(2)	−0.006(1)	0.004(1)	0.003(1)
C(8)	4a	0.0850(3)	0.5816(3)	0.8515(1)	0.038(1)	0.051(2)	0.043(1)	−0.007(1)	0.002(1)	0.004(1)
C(9)	4a	0.2412(3)	0.6676(3)	0.8532(2)	0.041(2)	0.063(2)	0.072(2)	−0.009(1)	0.002(1)	0.019(2)
C(10)	4a	0.2929(3)	0.4576(3)	0.8972(1)	0.035(1)	0.047(2)	0.058(2)	−0.006(1)	0.002(1)	0.001(1)
C(11)	4a	0.3510(4)	0.4208(3)	0.9622(2)	0.058(2)	0.062(2)	0.061(2)	0.010(2)	−0.012(2)	−0.009(2)
C(12)	4a	0.2809(5)	0.2845(3)	0.9819(2)	0.074(2)	0.082(2)	0.062(2)	0.011(2)	0.000(2)	0.014(2)
C(13)	4a	0.3213(5)	0.1754(3)	0.9349(2)	0.077(2)	0.054(2)	0.098(3)	0.006(2)	−0.015(2)	0.006(2)
C(14)	4a	0.2643(5)	0.2142(3)	0.8691(2)	0.073(2)	0.057(2)	0.083(2)	0.005(2)	−0.011(2)	−0.017(2)
C(15)	4a	0.3350(4)	0.3502(3)	0.8495(1)	0.053(2)	0.072(2)	0.056(2)	0.007(2)	0.007(1)	−0.005(2)

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