Crystal structure of (3*aS*,4*R*,5*S*,6*R*)-4-acetoxy-5,6-isopropylidenedioxy-3*a*,4,5,6-tetrahydro-3*H*-cyclopent[*c*]isoxazole, C₁₁H₁₅NO₅

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Abstract

C₁₁H₁₅NO₅, orthorhombic, *P*2₁2₁2₁ (No. 19), *a* = 5.5206(6) Å, *b* = 14.0083(9) Å, *c* = 15.6557(9) Å, *V* = 1210.7 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.052, *wR*_{ref}(*F*²) = 0.162, *T* = 293 K.

Source of material

The title compound has been obtained by intramolecular 1,3-dipolar nitrile oxide cycloaddition starting from the 4-*O*-acetyl-2,3-*O*-isopropylidene-*D*-lyxo-5-hexenose-oxime [1] by addition of *N*-chlorosuccinimide, a catalytic amount of triethylamine and small amounts of HCl gas in toluene [2–7]. The main diastereomer (ratio of diastereomers = 98:2) was separated by column chromatography using petroleum ether/ethyl acetate (1:1). Crystallization from ethyl acetate afforded the title compound in the form of colourless crystals (mp 436 K – 437 K; [*α*]₂₀^D = +154, *c* = 0.50, in CH₃OH), cf. [8].

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
N(1)	4 <i>a</i>	0.5632(7)	0.4096(2)	0.6108(2)	0.048(2)	0.060(2)	0.093(2)	−0.008(2)	−0.005(2)	−0.007(2)
O(1)	4 <i>a</i>	0.6585(7)	0.3394(2)	0.5526(2)	0.072(2)	0.062(2)	0.106(2)	−0.014(2)	0.000(2)	−0.019(2)
C(1)	4 <i>a</i>	0.8946(9)	0.3722(3)	0.5222(3)	0.064(3)	0.051(2)	0.078(2)	0.002(2)	−0.002(2)	−0.006(2)
O(2)	4 <i>a</i>	0.7965(5)	0.5509(2)	0.7621(2)	0.051(2)	0.073(2)	0.061(1)	−0.012(2)	0.003(1)	0.005(1)
C(2)	4 <i>a</i>	0.9693(6)	0.4477(2)	0.5855(2)	0.033(2)	0.045(2)	0.064(2)	0.003(2)	−0.004(2)	0.006(1)

Discussion

For crystal structure of a related isoxazoline see [8]. Determined by space group symmetry the packing of the molecules are in an antiparallel orientation. We observe also nonpolar channels made by the dimethyl groups of the dioxolan moieties along the *a*-axis.

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.1 × 0.1 × 0.75 mm
Wavelength:	Cu K _α radiation (1.54178 Å)
μ:	8.89 cm ^{−1}
Diffractometer, scan mode:	Siemens P4, ω
2θ _{max} :	135.94°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	1714, 1555
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 1240
<i>N</i> (<i>param</i>) _{refined} :	155
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(1A)	4 <i>a</i>	0.8820	0.3987	0.4651	0.077
H(1B)	4 <i>a</i>	1.0105	0.3201	0.5214	0.077
H(2)	4 <i>a</i>	1.0902	0.4222	0.6252	0.056
H(4)	4 <i>a</i>	0.5732	0.5921	0.6672	0.057
H(5)	4 <i>a</i>	0.8989	0.6763	0.6118	0.057
H(6)	4 <i>a</i>	1.2107	0.5569	0.5497	0.056
H(8A)	4 <i>a</i>	0.8265	0.7096	0.8540	0.137
H(8B)	4 <i>a</i>	0.8041	0.7359	0.7570	0.137
H(8C)	4 <i>a</i>	1.0484	0.7537	0.8055	0.137
H(9A)	4 <i>a</i>	1.0795	0.5679	0.8961	0.144
H(9B)	4 <i>a</i>	1.3046	0.6077	0.8465	0.144
H(9C)	4 <i>a</i>	1.2027	0.5062	0.8247	0.144
H(11A)	4 <i>a</i>	0.6850	0.6189	0.3634	0.122
H(11B)	4 <i>a</i>	0.9032	0.6549	0.3080	0.122
H(11C)	4 <i>a</i>	0.7577	0.7271	0.3648	0.122

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Table 3. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(3)	4a	1.1219(5)	0.6150(2)	0.6971(2)	0.042(2)	0.072(2)	0.053(1)	-0.011(1)	-0.002(1)	-0.002(1)
C(3)	4a	0.7351(7)	0.4648(2)	0.6305(2)	0.032(2)	0.050(2)	0.067(2)	-0.003(2)	-0.002(2)	0.005(2)
O(4)	4a	0.8994(5)	0.5722(2)	0.4833(2)	0.055(2)	0.052(1)	0.056(1)	-0.006(1)	-0.012(1)	0.009(1)
C(4)	4a	0.7280(7)	0.5589(2)	0.6748(2)	0.029(2)	0.053(2)	0.060(2)	0.002(2)	-0.001(2)	0.000(2)
O(5)	4a	1.1700(7)	0.6856(2)	0.4502(2)	0.085(3)	0.082(2)	0.101(2)	-0.032(2)	-0.002(2)	0.028(2)
C(5)	4a	0.9442(7)	0.6123(2)	0.6314(2)	0.042(2)	0.045(2)	0.056(2)	0.001(2)	-0.007(2)	0.000(1)
C(6)	4a	1.0360(7)	0.5495(2)	0.5588(2)	0.038(2)	0.052(2)	0.050(2)	-0.002(2)	-0.004(2)	0.002(1)
C(7)	4a	0.9956(8)	0.6130(3)	0.7765(2)	0.050(3)	0.074(2)	0.054(2)	-0.014(2)	0.004(2)	0.001(2)
C(8)	4a	0.911(1)	0.7121(3)	0.8004(3)	0.109(5)	0.079(3)	0.086(3)	-0.016(3)	0.024(4)	-0.024(2)
C(9)	4a	1.160(1)	0.5698(5)	0.8418(3)	0.075(4)	0.151(5)	0.063(2)	-0.006(4)	-0.016(3)	0.020(3)
C(10)	4a	0.9862(9)	0.6442(2)	0.4336(2)	0.074(3)	0.045(2)	0.059(2)	-0.002(2)	0.001(2)	0.007(2)
C(11)	4a	0.818(1)	0.6629(3)	0.3610(3)	0.119(5)	0.058(2)	0.068(2)	0.002(3)	-0.017(3)	0.012(2)

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