Crystal structure of (3*aS*,4*R*,5*S*,6*R*,6*aS*)-4,5,6-trihydroxy-5,6-*O*-isopropylidene-3,3*a*,4,5,6,6*a*-hexahydro-1*H*-cyclopent[*c*]isoxazole, C₉H₁₅NO₄

Z. Gültekin, W. Frey and V. Jäger*

Universität Stuttgart, Institut für Organische Chemie, Pfaffenwaldring 55, D-70569 Stuttgart, Germany

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O2–H2...O3 angle is about 174°. The H1...O4 distance is 2.24 Å and the N1–H1...O4 angle is about 170°. The nitrogen is strongly pyramidalized. In the packing diagram, we also observe polar channels of the hydrogen bond systems and nonpolar channels of the dimethyl groups, both along the *c*-axis.

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.07 × 0.15 × 0.35 mm
Wavelength:	Cu K α radiation (1.54178 Å)
μ :	8.85 cm ⁻¹
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{\max}$:	134.8°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	1456, 1294
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1052
$N(\text{param})_{\text{refined}}$:	133
Programs:	SHELXS-87 [7], SHELXL-97 [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(1)	4 <i>c</i>	−0.123(4)	0.711(2)	0.650(7)	0.05(1)
H(1A)	4 <i>c</i>	−0.2553	0.8324	1.0187	0.067
H(1B)	4 <i>c</i>	−0.3221	0.7812	0.8354	0.067
H(2)	4 <i>c</i>	−0.1778	0.9740	0.8689	0.101
H(2A)	4 <i>c</i>	−0.2347	0.8418	0.5241	0.051
H(3)	4 <i>c</i>	−0.0217	0.7870	0.4119	0.050
H(4)	4 <i>c</i>	0.1816	0.8017	0.7845	0.049
H(5)	4 <i>c</i>	0.0615	0.8784	1.0089	0.046
H(6)	4 <i>c</i>	−0.0803	0.9271	0.5716	0.053
H(8A)	4 <i>c</i>	0.4732	0.9319	0.7548	0.102
H(8B)	4 <i>c</i>	0.4884	0.8754	0.5671	0.102
H(8C)	4 <i>c</i>	0.4089	0.8654	0.8266	0.102
H(9A)	4 <i>c</i>	0.3355	0.9972	0.4346	0.097
H(9B)	4 <i>c</i>	0.1845	0.9716	0.3169	0.097
H(9C)	4 <i>c</i>	0.3411	0.9434	0.2338	0.097

Abstract

C₉H₁₅NO₄, orthorhombic, $P2_12_12$ (No. 18), $a = 8.8078(6)$ Å, $b = 21.209(1)$ Å, $c = 5.3420(4)$ Å, $V = 997.9$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.045$, $wR_{\text{ref}}(F^2) = 0.123$, $T = 293$ K.

Source of material

The title compound has been obtained by heating 2,3-*O*-isopropylidene-2,3,4-trihydroxy-D-lyxo-5-hexenose oxime [1] in pyridine [2–6]. The cycloaddition afforded one single diastereomer, which was purified by column chromatography (ethyl acetate). Crystallization from ethyl acetate gave colourless crystals (mp 405 K – 406 K; $[\alpha]_{20}^D = +8.4$, $c = 0.50$, MeOH).

Discussion

We observe a strong intermolecular hydrogen bond interaction, where the oxygen atoms (O3, O4) of the dioxolane moieties act as acceptors. The hydroxy group (O2) and N1–H1 are the donors of these systems. In the cell plot, it is obvious that the interaction between the oxygen atoms of the dioxolane group and the hydroxy function occurs pairwise. The H2...O3 distance is 2.0 Å and the

* Correspondence author (e-mail: jaeger.ioc@po.uni-stuttgart.de)

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)	4c	−0.1159(3)	0.7593(1)	0.9648(6)	0.068(2)	0.051(1)	0.059(2)	−0.013(1)	−0.011(2)	0.014(1)
N(1)	4c	−0.0464(3)	0.7367(1)	0.7338(7)	0.056(2)	0.032(1)	0.076(3)	−0.005(1)	−0.012(2)	−0.001(2)
C(1)	4c	−0.2305(4)	0.8033(2)	0.8848(9)	0.055(2)	0.047(2)	0.066(3)	−0.011(2)	0.004(2)	0.002(2)
O(2)	4c	−0.1852(3)	0.9361(1)	0.8959(6)	0.071(2)	0.038(1)	0.091(2)	0.008(1)	0.040(2)	−0.003(2)
C(2)	4c	−0.1621(4)	0.8384(2)	0.6624(7)	0.042(2)	0.042(2)	0.044(2)	0.005(1)	−0.004(2)	0.000(2)
O(3)	4c	0.1747(2)	0.93497(9)	0.7707(5)	0.051(1)	0.037(1)	0.050(2)	−0.007(1)	0.010(1)	−0.007(1)
C(3)	4c	−0.0257(4)	0.7951(1)	0.5924(8)	0.043(2)	0.037(2)	0.046(2)	0.001(1)	−0.006(2)	−0.005(2)
O(4)	4c	0.1981(3)	0.8571(1)	0.4781(5)	0.048(1)	0.039(1)	0.049(2)	−0.003(1)	0.005(1)	−0.011(1)
C(4)	4c	0.1165(4)	0.8292(1)	0.6835(7)	0.043(2)	0.033(1)	0.045(2)	0.002(1)	−0.005(2)	0.000(2)
C(5)	4c	0.0652(3)	0.8869(1)	0.8287(7)	0.046(2)	0.032(1)	0.038(2)	−0.007(1)	0.005(2)	−0.001(2)
C(6)	4c	−0.0915(4)	0.9025(1)	0.7257(8)	0.046(2)	0.036(1)	0.049(2)	0.008(1)	0.009(2)	0.004(2)
C(7)	4c	0.2738(4)	0.9119(2)	0.5764(7)	0.044(2)	0.039(2)	0.048(2)	−0.004(1)	−0.001(2)	−0.003(2)
C(8)	4c	0.4249(4)	0.8946(2)	0.692(1)	0.044(2)	0.064(2)	0.096(4)	−0.001(2)	−0.017(2)	−0.008(3)
C(9)	4c	0.2847(5)	0.9604(2)	0.3719(8)	0.083(3)	0.059(2)	0.053(2)	−0.020(2)	0.009(3)	0.003(2)

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