Crystal structure of (3a*S*,4*R*,5*S*,6*R*,6a*S*)-1-benzyl-4,5,6-trihydroxy-5,6-*O*-isopropylidene-3,3a,4,5,6,6a-hexahydro-1*H*-cyclopent[*c*]isoxazole, $C_{16}H_{21}NO_4$

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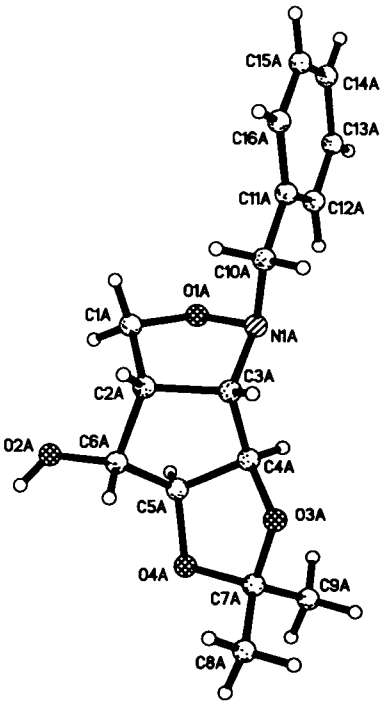

Abstract

$C_{16}H_{21}NO_4$, monoclinic, *C*121 (No. 5), $a = 39.820(2)$ Å, $b = 5.3038(3)$ Å, $c = 15.4373(9)$ Å, $\beta = 110.222(4)°$, $V = 3059.4$ Å^3, $Z = 8$, $R_{gt}(F) = 0.068$, $wR_{obs}(F^2) = 0.184$, $T = 293$ K.

Source of material

The title compound has been obtained by heating 2,3-*O*-isopropylidene-D-lyxo-5,6-dideoxy-5-hexenose [1] and *N*-benzylhydroxylamine in DMF [2-5]. After column chromatography, two separable regioisomers (ratio of regioisomers = 93:7) were isolated [6]. The major isomer was obtained in the form of colourless crystals by crystallization from ethyl acetate (mp 366 K – 367 K; $[\alpha]_{20}^{D} = +36$, c = 0.50, in $CHCl_3$).

Discussion

The title compound crystallizes with two independent molecules in the asymmetric unit. The crystal structure is stabilized by a network of intermolecular hydrogen bonds, where the hydroxy group of the molecules acts both as donor and acceptor. The H2A···O2B distance is 1.95 Å and the O2A–H2A···O2B angle is about 167°. One oxygen atom (O4) of the dioxolane moiety also functions as hydrogen bond acceptor. The H2B···O4 distance is 1.94 Å and the O2B–H2B···O4 angle is about 170°. In the packing diagram of the cell plot we observe layers of the benzyl moieties localized near the *ab*-face of the cell.

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Table 1. Data collection and handling.

Crystal:	colourless plates, size 0.05 × 0.15 × 0.8 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	7.43 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	136°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3745, 3572
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2675
$N(param)_{refined}$:	382
Programs:	SHELXS-86 [7], SHELXL-93 [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A1)	4*c*	0.1654(2)	0.829(2)	0.7088(4)	0.095
H(1A2)	4*c*	0.1831(2)	0.639(2)	0.7905(4)	0.095
H(2A)	4*c*	0.1016(3)	0.675(9)	0.536(5)	0.149
H(2A1)	4*c*	0.1638(1)	0.330(1)	0.6864(4)	0.081
H(3A)	4*c*	0.2144(1)	0.229(1)	0.6584(3)	0.072
H(4A)	4*c*	0.2284(1)	0.619(1)	0.5624(3)	0.062
H(5A)	4*c*	0.1755(1)	0.832(1)	0.5263(3)	0.068
H(6A)	4*c*	0.1361(1)	0.393(1)	0.5323(3)	0.079
H(8A1)	4*c*	0.1805(2)	0.085(2)	0.3330(4)	0.148
H(8A2)	4*c*	0.1489(2)	0.271(2)	0.2853(4)	0.148
H(8A3)	4*c*	0.1495(2)	0.118(2)	0.3728(4)	0.148
H(9A1)	4*c*	0.2257(2)	0.427(2)	0.3553(4)	0.122
H(9A2)	4*c*	0.2227(2)	0.671(2)	0.4093(4)	0.122
H(9A3)	4*c*	0.1959(2)	0.627(2)	0.3088(4)	0.122
H(10A)	4*c*	0.2595(2)	0.259(1)	0.8061(3)	0.086
H(10B)	4*c*	0.2282(2)	0.380(1)	0.8311(3)	0.086
H(12A)	4*c*	0.2900(2)	0.822(2)	0.8149(4)	0.086
H(13A)	4*c*	0.3307(2)	1.036(2)	0.9355(4)	0.100
H(14A)	4*c*	0.3424(2)	0.905(2)	1.0846(4)	0.111
H(15A)	4*c*	0.3120(2)	0.571(2)	1.1139(4)	0.113
H(16A)	4*c*	0.2705(2)	0.357(2)	0.9946(3)	0.097
H(1B1)	4*c*	–0.0687(1)	0.791(1)	0.6380(3)	0.068
H(1B2)	4*c*	–0.0928(1)	0.644(1)	0.6833(3)	0.068
H(2B)	4*c*	–0.043(1)	0.56(1)	0.479(2)	0.099
H(2B1)	4*c*	–0.0724(1)	0.286(1)	0.6413(3)	0.056
H(3B)	4*c*	–0.0272(1)	0.164(1)	0.7733(2)	0.056
H(4B)	4*c*	0.0249(1)	0.517(1)	0.8041(3)	0.055
H(5B)	4*c*	0.0019(1)	0.730(1)	0.6660(2)	0.054
H(6B)	4*c*	–0.0286(1)	0.271(1)	0.5781(2)	0.058
H(8B1)	4*c*	0.0746(2)	0.128(1)	0.6022(5)	0.126
H(8B2)	4*c*	0.0365(2)	0.019(1)	0.5891(5)	0.126
H(8B3)	4*c*	0.0699(2)	–0.070(1)	0.6724(5)	0.126

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(9B1)	4c	0.1013(1)	0.453(2)	0.7238(4)	0.136
H(9B2)	4c	0.0993(1)	0.272(2)	0.8023(4)	0.136
H(9B3)	4c	0.0807(1)	0.537(2)	0.7890(4)	0.136
H(10C)	4c	-0.0543(1)	0.264(1)	0.8936(3)	0.069
H(10D)	4c	-0.0825(1)	0.366(1)	0.8018(3)	0.069

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(12B)	4c	-0.0287(2)	0.608(2)	1.0224(3)	0.086
H(13B)	4c	-0.0454(2)	0.927(2)	1.1015(4)	0.100
H(14B)	4c	-0.1001(2)	1.118(2)	1.0386(5)	0.107
H(15B)	4c	-0.1382(2)	1.001(2)	0.8947(5)	0.104
H(16B)	4c	-0.1218(1)	0.686(2)	0.8157(4)	0.085

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1A)	4c	0.2174(1)	0.7750(9)	0.7344(2)	0.084(2)	0.053(2)	0.069(2)	0.007(2)	0.010(2)	-0.001(2)
N(1A)	4c	0.2370(1)	0.553(1)	0.7258(3)	0.074(2)	0.050(3)	0.052(2)	0.004(3)	0.011(2)	0.004(2)
C(1A)	4c	0.1827(2)	0.694(2)	0.7302(4)	0.085(3)	0.079(5)	0.079(3)	0.014(4)	0.035(3)	-0.015(4)
O(2A)	4c	0.12148(9)	0.723(1)	0.5693(3)	0.058(2)	0.116(5)	0.119(3)	0.009(3)	0.024(2)	-0.046(4)
C(2A)	4c	0.1731(1)	0.473(1)	0.6619(4)	0.077(3)	0.059(4)	0.076(3)	-0.012(3)	0.041(2)	-0.007(3)
O(3A)	4c	0.20348(9)	0.3077(8)	0.4940(2)	0.071(2)	0.055(2)	0.053(2)	0.014(2)	0.009(1)	-0.003(2)
C(3A)	4c	0.2096(1)	0.410(1)	0.6530(3)	0.070(3)	0.051(3)	0.059(2)	0.001(3)	0.019(2)	0.001(2)
C(4A)	4c	0.2079(1)	0.511(1)	0.5583(3)	0.050(2)	0.049(3)	0.052(2)	0.002(2)	0.012(2)	-0.005(2)
O(4A)	4c	0.16001(9)	0.5888(9)	0.4202(2)	0.061(2)	0.073(3)	0.066(2)	0.023(2)	0.010(1)	0.001(2)
C(5A)	4c	0.1724(1)	0.650(1)	0.5167(3)	0.053(2)	0.049(3)	0.065(2)	0.010(3)	0.016(2)	-0.008(2)
C(6A)	4c	0.1480(1)	0.545(1)	0.5647(3)	0.054(2)	0.069(4)	0.074(3)	-0.005(3)	0.023(2)	-0.017(3)
C(7A)	4c	0.1843(1)	0.412(1)	0.4042(3)	0.062(2)	0.052(3)	0.054(2)	0.016(3)	0.003(2)	0.000(2)
C(8A)	4c	0.1640(2)	0.203(2)	0.3433(4)	0.124(5)	0.064(4)	0.076(3)	0.003(5)	-0.006(3)	-0.020(4)
C(9A)	4c	0.2094(2)	0.546(2)	0.3659(4)	0.083(3)	0.091(5)	0.075(3)	0.015(4)	0.033(3)	0.012(4)
C(10A)	4c	0.2488(2)	0.418(1)	0.8139(3)	0.091(3)	0.060(4)	0.058(2)	0.002(4)	0.018(2)	0.012(3)
C(11A)	4c	0.2755(1)	0.564(1)	0.8906(3)	0.069(3)	0.061(4)	0.053(2)	0.006(3)	0.016(2)	-0.002(3)
C(12A)	4c	0.2941(2)	0.769(2)	0.8751(4)	0.074(3)	0.072(4)	0.064(3)	0.001(4)	0.019(2)	0.000(3)
C(13A)	4c	0.3187(2)	0.897(2)	0.9474(4)	0.072(3)	0.075(5)	0.095(4)	0.001(4)	0.018(3)	-0.009(4)
C(14A)	4c	0.3255(2)	0.821(2)	1.0362(4)	0.081(4)	0.101(6)	0.072(3)	0.032(5)	-0.004(3)	-0.017(4)
C(15A)	4c	0.3076(2)	0.622(2)	1.0533(4)	0.107(5)	0.104(7)	0.055(3)	0.033(5)	0.009(3)	0.002(4)
C(16A)	4c	0.2827(2)	0.493(2)	0.9815(3)	0.089(4)	0.088(5)	0.063(3)	0.015(4)	0.025(3)	0.010(3)
O(1B)	4c	-0.04485(9)	0.7381(7)	0.7741(2)	0.071(2)	0.038(2)	0.060(2)	-0.004(2)	0.031(1)	-0.003(2)
N(1B)	4c	-0.03291(9)	0.5082(8)	0.8264(2)	0.057(2)	0.043(2)	0.049(2)	0.000(2)	0.026(1)	-0.002(2)
C(1B)	4c	-0.0686(1)	0.665(1)	0.6836(3)	0.060(2)	0.052(3)	0.060(2)	0.012(3)	0.023(2)	0.002(2)
O(2B)	4c	-0.05095(8)	0.590(1)	0.5204(2)	0.063(2)	0.089(3)	0.048(1)	0.026(2)	0.023(1)	0.014(2)
C(2B)	4c	-0.0536(1)	0.414(1)	0.6633(3)	0.046(2)	0.045(3)	0.049(2)	-0.005(2)	0.016(2)	-0.010(2)
O(3B)	4c	0.02959(8)	0.1946(8)	0.7387(2)	0.061(2)	0.054(2)	0.073(2)	0.016(2)	0.035(1)	0.020(2)
C(3B)	4c	-0.0259(1)	0.343(1)	0.7587(2)	0.052(2)	0.045(3)	0.047(2)	-0.001(2)	0.023(2)	0.001(2)
O(4B)	4c	0.03242(7)	0.4757(7)	0.6306(2)	0.053(2)	0.055(2)	0.055(1)	0.013(2)	0.027(1)	0.012(2)
C(4B)	4c	0.0105(1)	0.413(1)	0.7518(3)	0.047(2)	0.046(3)	0.046(2)	0.001(2)	0.018(1)	0.000(2)
C(5B)	4c	0.0032(1)	0.547(1)	0.6596(2)	0.049(2)	0.044(3)	0.049(2)	0.002(2)	0.024(2)	0.001(2)
C(6B)	4c	-0.0325(1)	0.439(1)	0.5990(2)	0.048(2)	0.054(3)	0.042(2)	0.004(2)	0.013(1)	0.002(2)
C(7B)	4c	0.0527(1)	0.282(1)	0.6923(3)	0.053(2)	0.055(3)	0.064(2)	0.015(3)	0.029(2)	0.012(2)
C(8B)	4c	0.0590(2)	0.070(1)	0.6336(5)	0.114(4)	0.055(4)	0.114(4)	0.025(4)	0.078(4)	0.011(4)
C(9B)	4c	0.0866(1)	0.397(2)	0.7578(4)	0.053(2)	0.134(8)	0.075(3)	-0.005(4)	0.011(2)	0.023(4)
C(10B)	4c	-0.0623(1)	0.413(1)	0.8557(3)	0.062(2)	0.055(3)	0.063(2)	-0.003(3)	0.031(2)	-0.004(3)
C(11B)	4c	-0.0736(1)	0.612(1)	0.9098(3)	0.064(2)	0.063(4)	0.053(2)	-0.001(3)	0.033(2)	-0.003(2)
C(12B)	4c	-0.0508(2)	0.686(2)	0.9962(3)	0.086(3)	0.080(5)	0.051(2)	0.010(4)	0.027(2)	-0.003(3)
C(13B)	4c	-0.0609(2)	0.877(2)	1.0437(4)	0.115(5)	0.083(5)	0.056(2)	0.012(5)	0.034(3)	-0.012(3)
C(14B)	4c	-0.0934(2)	0.992(2)	1.0060(5)	0.120(5)	0.073(5)	0.096(4)	0.012(5)	0.064(4)	-0.008(4)
C(15B)	4c	-0.1162(2)	0.922(2)	0.9206(5)	0.084(4)	0.074(5)	0.118(5)	0.020(4)	0.053(4)	0.002(4)
C(16B)	4c	-0.1062(1)	0.733(2)	0.8737(4)	0.061(2)	0.078(5)	0.080(3)	0.000(3)	0.031(2)	-0.013(3)

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