Crystal structure of (1*S*,2*R*,3*S*,4*S*,5*R*)-7-benzyl-2,3-*O*-isopropylidene-6-oxa-7-aza-bicyclo[3.2.1]octan-2,3,4-triol, $C_{16}H_{21}NO_4$

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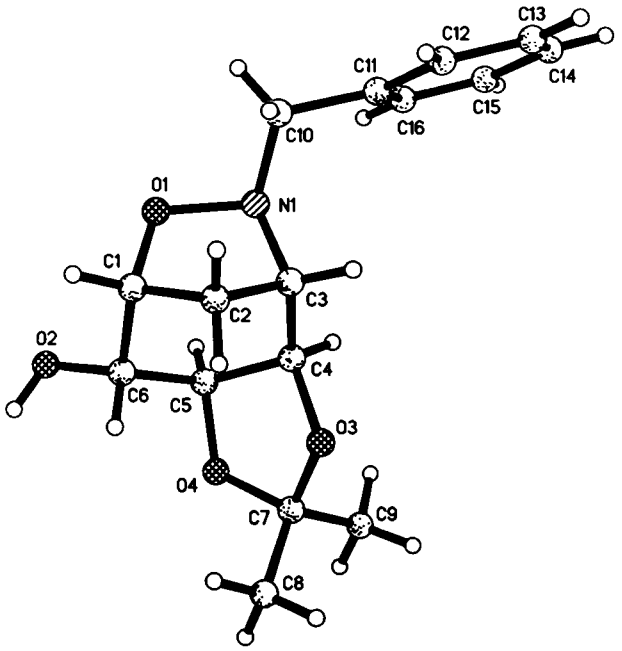

Abstract

$C_{16}H_{21}NO_4$, monoclinic, *C*121 (No. 5), $a = 21.1727(8)$ Å, $b = 5.5793(3)$ Å, $c = 12.8757(6)$ Å, $\beta = 93.320(4)°$, $V = 1518.4$ Å^3, $Z = 4$, $R_{gt}(F) = 0.055$, $wR_{ref}(F^2) = 0.169$, $T = 293$ K.

Source of material

The title compound has been obtained as a by-product by heating 2,3-*O*-isopropylidene-D-lyxo-5,6-dideoxy-5-hexenose [1] and *N*-benzyl-hydroxylamine in DMF [2-5] which was separated from the main product (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 (ratio of regioisomers = 93:7) by column chromatography using petroleum ether/ethyl acetate (20:80) [6]. Crystallization from ethyl acetate afforded the title compound in the form of colourless crystals (mp 399 K; $[\alpha]_{20}^{D} = -179$, c = 0.50, in CH_3OH).

Discussion

We observe a pairwise intermolecular hydrogen bond interaction between the hydroxy group which works as donor and one of the oxygens of the dioxolane group as acceptor. The O2A···O4 distance is 2.97 Å and the O2–H2···O4 angle of 166° shows the nearly linear interaction. In the unit cell plot we observe non-polar layers in the *bc*-plane built by the benzyl moieties and the methyl groups. There exists also polar channels along the *b*-axis where the hydrogen bond interactions are localized.

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.1 × 0.1 × 0.5 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	7.48 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	135.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1938, 1795
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1464
$N(param)_{refined}$:	192
Programs:	SHELXS-87 [7], SHELXL-97 [8]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*c*	0.6751	0.5029	–0.0359	0.072
H(2)	4*c*	0.5473	0.2631	–0.0594	0.147
H(2A)	4*c*	0.6492	0.8267	0.0903	0.079
H(2B)	4*c*	0.7222	0.7720	0.0812	0.079
H(3)	4*c*	0.7058	0.6942	0.2588	0.063
H(4)	4*c*	0.6235	0.4302	0.3027	0.057
H(5)	4*c*	0.5911	0.1955	0.1702	0.060
H(6)	4*c*	0.5710	0.5641	0.0213	0.072
H(8A)	4*c*	0.4323	0.7673	0.1768	0.102
H(8B)	4*c*	0.4901	0.8441	0.1136	0.102
H(8C)	4*c*	0.4781	0.9697	0.2196	0.102
H(9A)	4*c*	0.4506	0.5222	0.3380	0.140
H(9B)	4*c*	0.4962	0.7171	0.3878	0.140
H(9C)	4*c*	0.5200	0.4530	0.3753	0.140
H(10A)	4*c*	0.8000	0.5370	0.1643	0.078
H(10B)	4*c*	0.8088	0.2576	0.1704	0.078
H(12)	4*c*	0.8597	0.7207	0.3016	0.088
H(13)	4*c*	0.8954	0.7522	0.4750	0.106
H(14)	4*c*	0.8696	0.4636	0.5905	0.101
H(15)	4*c*	0.8061	0.1451	0.5362	0.109
H(16)	4*c*	0.7702	0.1157	0.3653	0.095

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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(1)	4c	0.7042(1)	0.2956(6)	0.0840(2)	0.065(2)	0.057(2)	0.052(1)	0.009(2)	–0.004(1)	–0.011(1)
N(1)	4c	0.7196(1)	0.3623(7)	0.1937(2)	0.049(2)	0.066(3)	0.046(1)	0.003(2)	0.001(1)	–0.008(2)
C(1)	4c	0.6653(2)	0.4813(9)	0.0370(3)	0.076(2)	0.058(3)	0.046(2)	0.014(2)	0.009(2)	0.003(2)
O(2)	4c	0.5779(2)	0.2257(9)	–0.0209(3)	0.109(3)	0.086(3)	0.094(2)	0.023(3)	–0.049(2)	–0.045(3)
C(2)	4c	0.6819(2)	0.705(1)	0.0983(3)	0.081(3)	0.048(3)	0.070(2)	–0.002(2)	0.022(2)	0.003(2)
O(3)	4c	0.5796(1)	0.7214(6)	0.2458(2)	0.046(1)	0.060(2)	0.059(1)	–0.005(1)	0.005(1)	–0.012(2)
C(3)	4c	0.6847(2)	0.5902(9)	0.2062(3)	0.046(2)	0.055(3)	0.056(2)	–0.003(2)	0.006(1)	–0.014(2)
O(4)	4c	0.5176(1)	0.4200(6)	0.1765(2)	0.043(1)	0.055(2)	0.092(2)	0.001(1)	–0.006(1)	–0.010(2)
C(4)	4c	0.6199(2)	0.5161(9)	0.2363(3)	0.041(2)	0.057(3)	0.045(2)	0.002(2)	–0.001(1)	0.001(2)
C(5)	4c	0.5828(2)	0.3657(8)	0.1565(3)	0.045(2)	0.041(2)	0.063(2)	0.002(2)	–0.005(1)	–0.001(2)
C(6)	4c	0.5960(2)	0.424(1)	0.0438(3)	0.066(2)	0.060(3)	0.052(2)	0.012(2)	–0.013(2)	–0.013(2)
C(7)	4c	0.5156(2)	0.6375(9)	0.2385(3)	0.042(2)	0.060(3)	0.060(2)	–0.004(2)	0.003(2)	–0.003(2)
C(8)	4c	0.4754(2)	0.821(1)	0.1821(4)	0.065(2)	0.058(3)	0.081(3)	0.008(2)	–0.005(2)	–0.003(3)
C(9)	4c	0.4936(2)	0.577(2)	0.3445(4)	0.074(3)	0.134(6)	0.075(3)	–0.010(4)	0.023(2)	0.019(4)
C(10)	4c	0.7886(2)	0.394(1)	0.2016(3)	0.043(2)	0.092(4)	0.060(2)	0.005(2)	0.006(1)	–0.011(3)
C(11)	4c	0.8113(2)	0.415(1)	0.3149(3)	0.048(2)	0.071(3)	0.057(2)	0.001(2)	0.004(2)	–0.001(2)
C(12)	4c	0.8487(2)	0.604(1)	0.3487(4)	0.076(3)	0.077(4)	0.066(2)	–0.016(3)	–0.005(2)	–0.001(3)
C(13)	4c	0.8703(3)	0.623(1)	0.4529(4)	0.089(3)	0.095(5)	0.077(3)	–0.017(3)	–0.015(3)	–0.008(3)
C(14)	4c	0.8548(3)	0.453(1)	0.5213(4)	0.091(3)	0.093(5)	0.066(3)	0.009(4)	–0.015(2)	–0.001(3)
C(15)	4c	0.8170(3)	0.262(2)	0.4888(4)	0.109(4)	0.094(5)	0.069(3)	0.003(4)	–0.007(3)	0.014(3)
C(16)	4c	0.7956(2)	0.245(1)	0.3867(4)	0.082(3)	0.073(4)	0.081(3)	–0.013(3)	–0.003(2)	0.006(3)

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