Crystal structure of (+)-(3a*S*,6a*S*,1′*R*)-6-oxo-3-(1′-*O*-benzyl-1′,2′-dihydroxyethyl)-3a,4,6a-tetrahydrofuro[3,4-*d*]isoxazole, $C_{14}H_{15}NO_5$

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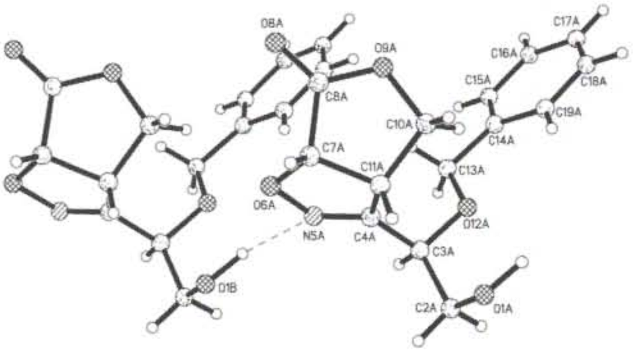

Abstract

$C_{14}H_{15}NO_5$, orthorhombic, $P2_12_12_1$ (No. 19), a = 6.232(1) Å, b = 8.444(1) Å, c = 26.141(4) Å, V = 1375.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.060, $wR(F^2)$ = 0.135, T = 293 K.

Source of material

The title compound [1] was prepared by 1,3-dipolar nitrile oxide cycloaddition of the corresponding hydroximoyl chloride [2, 3] with 2-butene-4-olide using NEt_3 as base. The reaction gave a mixture of regioisomers (4-oxo/6-oxo 83:17, d.r. ca. 1:1 each). The isomers were separated by MPLC. Crystallization from EtOAc/petrol ether gave the title compound in the form of colourless crystals, (mp 358 K – 359 K, $[\alpha]_D^{20}$ = +79.6, c = 0.885, $CHCl_3$).

Discussion

For crystal structures of related furoisoxazoles see [4-6]. An intermolecular hydrogen bond between the hydroxy group and N5 is observed. The distance O1—N5 is 2.847 Å and the O1-N1-N5 angle is 138°. We observe planes of the non-polar benzyl groups along the c-axis alternating with planes of the polar moiety of the molecule.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.25 × 0.4 × 0.8 mm
Wavelength:	Mo K_α radiation (0.71073 A)
µ:	1.02 cm^{-1}
Diffractometer, scan mode:	Nicolet P3, Wyckoff
$2\theta_{max}$:	54.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1851, 1851
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1383
$N(param)_{refined}$:	182
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(1)	4*a*	1.1803	0.2589	0.8274	0.108
H(2A)	4*a*	0.8281(7)	0.4481(4)	0.8065(2)	0.082
H(2B)	4*a*	0.9856(7)	0.4652(4)	0.8529(2)	0.082
H(3)	4*a*	0.6693(6)	0.3392(4)	0.8785(1)	0.062
H(7)	4*a*	0.5801(6)	−0.0770(4)	0.7400(1)	0.063
H(10A)	4*a*	1.0266(7)	−0.1285(4)	0.7975(2)	0.069
H(10B)	4*a*	0.9778(7)	−0.0508(4)	0.8511(2)	0.069
H(11)	4*a*	0.8522(6)	0.0827(4)	0.7655(1)	0.052
H(13A)	4*a*	0.6825(7)	0.0718(6)	0.9278(2)	0.086
H(13B)	4*a*	0.7202(7)	0.2304(6)	0.9579(2)	0.086
H(15)	4*a*	0.7196(9)	0.0639(6)	1.0358(2)	0.093
H(16)	4*a*	0.924(1)	−0.0831(6)	1.0930(2)	0.116
H(17)	4*a*	1.256(1)	−0.1717(7)	1.0699(3)	0.121
H(18)	4*a*	1.392(1)	−0.1163(6)	0.9898(2)	0.117
H(19)	4*a*	1.1914(8)	0.0272(6)	0.9322(2)	0.090

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)	4*a*	1.0795(5)	0.3123(4)	0.8019(1)	0.057(2)	0.074(2)	0.085(2)	0.004(2)	0.003(2)	0.019(2)
C(2)	4*a*	0.9190(7)	0.3895(4)	0.8299(2)	0.054(2)	0.042(2)	0.110(3)	−0.004(2)	0.007(3)	−0.012(2)
C(3)	4*a*	0.7802(6)	0.2770(4)	0.8610(1)	0.048(2)	0.049(2)	0.058(2)	0.016(2)	0.000(2)	−0.013(2)
C(4)	4*a*	0.6710(5)	0.1533(4)	0.8286(1)	0.042(2)	0.042(2)	0.041(2)	0.012(2)	0.002(2)	−0.002(2)
N(5)	4*a*	0.4674(5)	0.1521(4)	0.8270(1)	0.047(2)	0.059(2)	0.058(2)	0.011(2)	−0.002(2)	−0.008(2)
O(6)	4*a*	0.3963(4)	0.0361(4)	0.7919(1)	0.045(2)	0.075(2)	0.083(2)	0.004(2)	−0.012(2)	−0.025(2)
C(7)	4*a*	0.5785(6)	−0.0566(4)	0.7769(1)	0.050(2)	0.056(2)	0.051(2)	0.004(2)	−0.006(2)	−0.013(2)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(8)	$4a$	0.5833(8)	−0.2082(5)	0.8075(2)	0.073(3)	0.052(2)	0.062(2)	−0.006(3)	−0.002(3)	−0.012(2)
O(8)	$4a$	0.4430(7)	−0.3048(4)	0.8112(1)	0.109(3)	0.080(2)	0.103(2)	−0.041(3)	0.002(2)	−0.012(2)
O(9)	$4a$	0.7681(6)	−0.2214(3)	0.8322(1)	0.087(2)	0.046(1)	0.079(2)	0.006(2)	−0.014(2)	0.007(1)
C(10)	$4a$	0.9133(7)	−0.0926(4)	0.8202(2)	0.057(2)	0.048(2)	0.067(2)	0.017(2)	−0.012(2)	−0.015(2)
C(11)	$4a$	0.7771(6)	0.0328(4)	0.7942(1)	0.045(2)	0.046(2)	0.040(2)	0.004(2)	0.000(2)	−0.004(2)
O(12)	$4a$	0.9112(4)	0.2038(3)	0.89885(9)	0.047(1)	0.071(2)	0.054(1)	0.008(2)	−0.003(1)	−0.014(1)
C(13)	$4a$	0.7917(7)	0.1437(6)	0.9405(2)	0.064(3)	0.095(3)	0.057(2)	0.016(3)	0.006(2)	−0.001(2)
C(14)	$4a$	0.9330(8)	0.0586(5)	0.9773(2)	0.072(3)	0.062(2)	0.053(2)	−0.001(3)	−0.010(2)	−0.016(2)
C(15)	$4a$	0.8541(9)	0.0268(6)	1.0262(2)	0.095(4)	0.075(3)	0.062(2)	−0.016(3)	−0.011(3)	−0.010(2)
C(16)	$4a$	0.977(1)	−0.0606(6)	1.0606(2)	0.145(6)	0.070(3)	0.073(3)	−0.042(4)	−0.040(4)	0.006(3)
C(17)	$4a$	1.175(1)	−0.1132(7)	1.0468(3)	0.146(6)	0.060(3)	0.096(4)	−0.009(4)	−0.062(4)	0.000(3)
C(18)	$4a$	1.256(1)	−0.0805(6)	0.9988(2)	0.110(4)	0.077(3)	0.105(4)	0.028(4)	−0.046(4)	−0.029(3)
C(19)	$4a$	1.1358(8)	0.0051(6)	0.9644(2)	0.080(3)	0.077(3)	0.070(3)	0.018(3)	−0.020(2)	−0.023(2)

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