Crystal structure of (5*S*,8*S*)-8-hydroxymethyl-1-methyl-2,7-dioxa-1-aza-spiro[4,4]nonan-6-one, $C_8H_{13}NO_4$

W. Frey, M. Imerhasan, Y. Bathich and V. Jäger*

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

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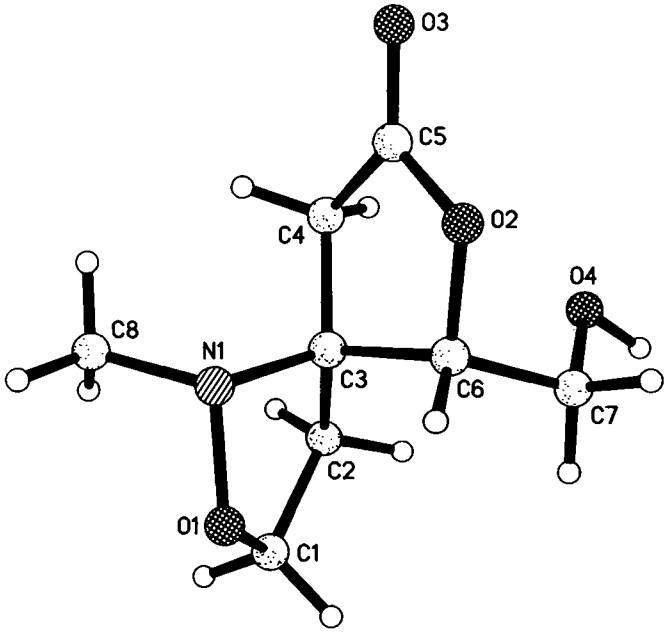

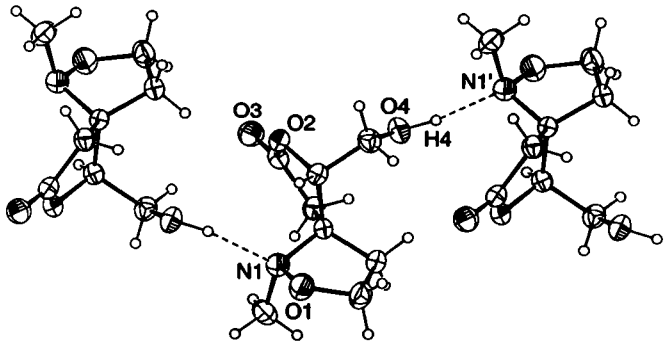

Abstract

$C_8H_{13}NO_4$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 8.8739(5)$ Å, $b = 9.5664(5)$ Å, $c = 10.7489(6)$ Å, $V = 912.5$ Å^3, $Z = 4$, $R_{gt}(F) = 0.048$, $wR_{ref}(F^2) = 0.134$, $T = 293$ K.

Source of material

The title compound has been obtained by 1,3-dipolar cycloaddition of ethylene and the nitrile oxide generated in situ from the hydroximoyl chloride derived from 2,3-*O*-cyclohexylidene-D-glyceraldehyde, followed by *N*-methylation of the intermediate isoxazoline with trimethyloxonium tetrafluoroborate [1-5]. Addition of lithium ethyl acetate to the isoxazolinium salt gave a mixture separated by MPLC (ratio of diastereoisomers 77:23). The major diastereomer was hydrolyzed using aqueous HCl to obtain the furanone [5,6]. Recrystallization from ethanol and petroleum ether afforded the title furanone in the form of colorless crystals (m.p. 383-384 K, $\alpha_D^{20} = -68.9°$, $c = 0.93$, CH_2Cl_2).

Discussion

Both five-membered ring systems have an envelope conformation, where the nitrogen N1 of the isoxazoline moiety and the "spirocarbon" of the furanone system are out of plane (figure, top). The molecules are arranged in chains along the *c* axis in two orientations (180°) with C-8 atoms (chemical numbering, corresponding to C6 of structural numbering) superposing. In the chains the molecules are connected to each other by intermolecular hydrogen bonds, where the hydroxy function acts as donor and the nitrogen with pronounced pyramidal coordination as acceptor (figure, bottom). The H4···N1 distance is 1.99 Å and the O4–H4···N1′ angle is 171°.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.2 × 0.4 × 0.4 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	9.28 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	135.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1640, 1389
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1304
$N(param)_{refined}$:	171
Programs:	SHELXS-97 [7], SHELXL-97 [8]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	4*a*	0.620(4)	0.278(4)	0.639(4)	0.09(1)
H(1B)	4*a*	0.443(5)	0.232(5)	0.675(4)	0.10(1)
H(2A)	4*a*	0.582(5)	0.500(4)	0.668(3)	0.07(1)
H(2B)	4*a*	0.409(5)	0.470(5)	0.724(4)	0.08(1)
H(4)	4*a*	0.127(5)	0.621(4)	0.781(4)	0.09(1)
H(4A)	4*a*	0.416(5)	0.720(5)	0.585(5)	0.10(2)
H(4B)	4*a*	0.517(3)	0.697(3)	0.472(3)	0.056(8)
H(6)	4*a*	0.222(4)	0.390(3)	0.482(4)	0.07(1)
H(7A)	4*a*	0.039(4)	0.498(3)	0.617(3)	0.052(8)
H(7B)	4*a*	0.176(4)	0.424(4)	0.697(3)	0.064(9)
H(8A)	4*a*	0.678(5)	0.368(5)	0.360(4)	0.09(1)
H(8B)	4*a*	0.706(4)	0.470(4)	0.484(4)	0.08(1)
H(8C)	4*a*	0.637(5)	0.555(5)	0.360(5)	0.10(2)

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

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}
N(1)	4*a*	0.4822(3)	0.4246(3)	0.4365(2)	0.057(1)	0.057(1)	0.043(1)	0.009(1)	0.002(1)	–0.007(1)
O(1)	4*a*	0.4800(3)	0.2877(2)	0.4946(2)	0.088(2)	0.052(1)	0.059(1)	0.015(1)	–0.009(1)	–0.007(1)
C(1)	4*a*	0.5118(5)	0.3056(4)	0.6271(3)	0.095(3)	0.063(2)	0.052(2)	0.022(2)	–0.011(2)	–0.001(2)
C(2)	4*a*	0.4835(3)	0.4587(3)	0.6554(3)	0.054(1)	0.061(2)	0.040(2)	0.004(1)	–0.005(1)	–0.001(1)
O(2)	4*a*	0.1929(2)	0.5969(2)	0.4338(2)	0.0482(9)	0.067(1)	0.040(1)	0.0045(9)	–0.0045(8)	0.0014(9)
O(3)	4*a*	0.2727(3)	0.8015(3)	0.3638(2)	0.075(2)	0.073(2)	0.063(2)	0.017(1)	0.009(1)	0.021(1)
C(3)	4*a*	0.4130(3)	0.5123(3)	0.5351(2)	0.045(1)	0.048(1)	0.035(1)	0.004(1)	0.001(1)	–0.004(1)
O(4)	4*a*	0.1732(3)	0.6310(2)	0.6996(2)	0.062(1)	0.066(1)	0.047(1)	–0.006(1)	0.010(1)	–0.006(1)
C(4)	4*a*	0.4277(3)	0.6675(3)	0.5078(3)	0.050(1)	0.052(2)	0.049(2)	–0.003(1)	–0.002(1)	–0.001(2)
C(5)	4*a*	0.2947(3)	0.6999(3)	0.4277(3)	0.054(1)	0.057(2)	0.040(2)	0.007(1)	0.007(1)	–0.001(2)
C(6)	4*a*	0.2405(3)	0.4902(3)	0.5227(3)	0.048(1)	0.052(2)	0.043(2)	0.001(1)	–0.004(1)	–0.001(1)
C(7)	4*a*	0.1443(3)	0.5025(3)	0.6383(3)	0.049(1)	0.060(2)	0.051(2)	–0.008(1)	0.006(1)	0.001(1)
C(8)	4*a*	0.6405(4)	0.4579(5)	0.4068(4)	0.059(2)	0.097(3)	0.060(2)	0.006(2)	0.009(2)	–0.018(2)

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