Crystal structure of methyl-5-amino-6,7-*O*-cyclohexylidene-5-deoxy-3*O*,5*N*-ethylidene- α -D-*ido*-L-*glycero*-hepto-furanoside, C₁₆H₂₇NO₆

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Table 1. Parameters used for the X-ray data collection

Crystal:	colorless block, size 0.4 x 0.4 x 1.0 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.97 cm ⁻¹
Diffractometer:	Nicolet P3
Scan mode:	Wyckoff
T _{measurement} :	293 K
2 θ _{max} :	55°
N(hkl) _{unique} :	2113
Criterion for I _o :	I _o > 2 σ (I _o)
N(param) _{refined} :	210
Programs:	SHELXS-86, SHELXL-93

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U _{iso}
H(1A)	4a	-0.1245	0.3172	0.2779	0.048
H(1)	4a	-0.0838(3)	0.3154(2)	0.4504(2)	0.043
H(2)	4a	0.2047(3)	0.3512(2)	0.4433(2)	0.045
H(3)	4a	0.2128(4)	0.1942(2)	0.4232(2)	0.050
H(4)	4a	0.4184(4)	0.2020(2)	0.2899(2)	0.054
H(5)	4a	0.4805(4)	0.3827(2)	0.3258(2)	0.053
H(6)	4a	-0.0900(4)	0.4663(2)	0.3351(2)	0.052
H(6A)	4a	0.612(2)	0.260(3)	0.386(1)	0.104
H(7A)	4a	0.0125(4)	0.4699(2)	0.5094(2)	0.064
H(7B)	4a	0.0929(4)	0.5290(2)	0.4338(2)	0.064
H(9A)	4a	-0.5277(5)	0.5665(2)	0.3931(2)	0.078
H(9B)	4a	-0.3584(5)	0.6158(2)	0.3587(2)	0.078
H(10A)	4a	-0.3640(6)	0.7217(2)	0.4708(3)	0.101
H(10B)	4a	-0.5489(6)	0.7231(2)	0.4267(3)	0.101
H(11A)	4a	-0.5778(6)	0.7194(3)	0.5758(3)	0.102
H(11B)	4a	-0.6647(6)	0.6314(3)	0.5344(3)	0.102
H(12A)	4a	-0.4944(6)	0.5854(3)	0.6497(3)	0.096
H(12B)	4a	-0.3295(6)	0.6348(3)	0.6105(3)	0.096
H(13A)	4a	-0.3087(6)	0.4772(2)	0.5817(2)	0.076
H(13B)	4a	-0.4958(6)	0.4769(2)	0.5399(2)	0.076
H(14A)	4a	0.4584(7)	0.4250(3)	0.1160(3)	0.161
H(14B)	4a	0.5485(7)	0.4565(3)	0.2014(3)	0.161
H(14C)	4a	0.3465(7)	0.4714(3)	0.1885(3)	0.161
H(15)	4a	-0.1017(4)	0.1602(2)	0.3726(2)	0.056
H(16A)	4a	-0.2853(5)	0.1467(3)	0.2519(3)	0.110
H(16B)	4a	-0.1204(5)	0.0826(3)	0.2427(3)	0.110
H(16C)	4a	-0.1290(5)	0.1767(3)	0.1928(3)	0.110

Source of material: The title compound (alternative name: (2*R*,4*R*,4*aS*,6*S*,7*S*,7*aR*)-7-hydroxy-4-[(1*R*)-1,2-*O*-cyclohexyldenedioxyethyl]-6-methoxy-2-methyl-perhydrofuro[2,3-*e*]-1,3-oxazine) was prepared by reduction of the corresponding isoxazoline (see ref. 1–4) using LiAlH₄, with subsequent protection of the resulting 1,3-aminoalcohol. Crystallization from petroleum ether/ethyl acetate (4:1) furnished the title compound in the form of colorless crystals [mp 427 K – 429 K, $[\alpha]_{20}^D = +41.8$ ($c = 0.52$, CHCl₃)].

The oxazine ring has chair conformation. The nitrogen atom is strongly pyramidalized and forms a hydrogen bridge to the 7-OH group of a neighbouring molecule. The cyclohexylidene groups are stacked in the crystal so that they lie on a directional vector connecting the centres of the rings.

C₁₆H₂₇NO₆, orthorhombic, $P2_12_12_1$ (No. 19), $a = 7.622(1)$ Å, $b = 14.432(2)$ Å, $c = 15.621(2)$ Å, $V = 1718.3$ Å³, $Z = 4$, $R(F) = 0.047$, $R_w(F^2) = 0.105$.

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	4a	0.1097(3)	0.1900(1)	0.3053(1)	0.034(1)	0.052(1)	0.061(1)	0.002(1)	-0.001(1)	-0.010(1)
N(1)	4a	-0.1401(3)	0.2879(2)	0.3286(2)	0.033(1)	0.045(1)	0.043(1)	-0.002(1)	-0.005(1)	-0.002(1)
C(1)	4a	-0.0473(3)	0.3407(2)	0.3950(2)	0.029(1)	0.048(1)	0.030(1)	-0.003(1)	0.002(1)	-0.000(1)
O(2)	4a	-0.2832(3)	0.4476(2)	0.4184(2)	0.035(1)	0.064(1)	0.090(2)	0.002(1)	-0.006(1)	-0.040(1)

Table 3. (Continued)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(2)	4a	0.1517(3)	0.3301(2)	0.3896(2)	0.030(1)	0.049(2)	0.032(1)	-0.005(1)	-0.002(1)	0.001(1)
O(3)	4a	-0.1400(3)	0.5735(2)	0.4722(2)	0.048(1)	0.051(1)	0.085(2)	-0.002(1)	-0.006(1)	-0.026(1)
C(3)	4a	0.2108(4)	0.2322(2)	0.3712(2)	0.031(1)	0.051(2)	0.043(1)	0.001(1)	-0.000(1)	0.007(1)
O(4)	4a	0.2202(3)	0.3836(1)	0.3201(1)	0.037(1)	0.055(1)	0.054(1)	0.0000(9)	0.011(1)	0.014(1)
C(4)	4a	0.3937(4)	0.2468(2)	0.3355(2)	0.031(1)	0.059(2)	0.046(1)	0.002(1)	0.002(1)	0.003(1)
C(5)	4a	0.3878(4)	0.3453(2)	0.2995(2)	0.032(1)	0.067(2)	0.035(1)	-0.007(1)	0.002(1)	0.001(1)
O(5)	4a	0.4106(4)	0.3432(2)	0.2127(1)	0.095(2)	0.091(2)	0.040(1)	0.013(2)	0.020(1)	0.010(1)
C(6)	4a	-0.1044(4)	0.4412(2)	0.3929(2)	0.039(1)	0.049(2)	0.042(1)	-0.000(1)	-0.004(1)	-0.008(1)
O(6)	4a	0.5175(3)	0.2388(2)	0.4019(2)	0.032(1)	0.109(2)	0.067(1)	-0.007(1)	-0.006(1)	0.031(2)
C(7)	4a	-0.0145(4)	0.5033(2)	0.4571(2)	0.039(2)	0.060(2)	0.062(2)	-0.003(2)	-0.004(2)	-0.017(2)
C(8)	4a	-0.3097(4)	0.5337(2)	0.4618(2)	0.043(2)	0.041(2)	0.054(2)	0.003(1)	-0.003(1)	-0.011(1)
C(9)	4a	-0.4212(5)	0.5983(2)	0.4102(2)	0.062(2)	0.067(2)	0.066(2)	0.003(2)	-0.009(2)	0.009(2)
C(10)	4a	-0.4692(6)	0.6856(2)	0.4605(3)	0.077(3)	0.041(2)	0.134(4)	0.010(2)	-0.020(3)	0.008(2)
C(11)	4a	-0.5540(6)	0.6627(3)	0.5445(3)	0.059(2)	0.066(2)	0.129(4)	0.014(2)	0.005(3)	-0.034(3)
C(12)	4a	-0.4363(6)	0.6015(3)	0.5966(3)	0.082(3)	0.086(3)	0.071(2)	0.007(2)	0.016(2)	-0.019(2)
C(13)	4a	-0.3903(6)	0.5135(2)	0.5479(2)	0.075(2)	0.052(2)	0.062(2)	0.000(2)	0.002(2)	0.006(2)
C(14)	4a	0.4436(7)	0.4310(3)	0.1768(3)	0.127(4)	0.117(4)	0.080(3)	0.025(3)	0.049(3)	0.052(3)
C(15)	4a	-0.0745(4)	0.1945(2)	0.3201(2)	0.035(1)	0.044(2)	0.061(2)	-0.003(1)	-0.001(1)	-0.002(2)
C(16)	4a	-0.1602(5)	0.1456(3)	0.2450(3)	0.050(2)	0.064(2)	0.106(3)	0.002(2)	-0.018(2)	-0.035(2)

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