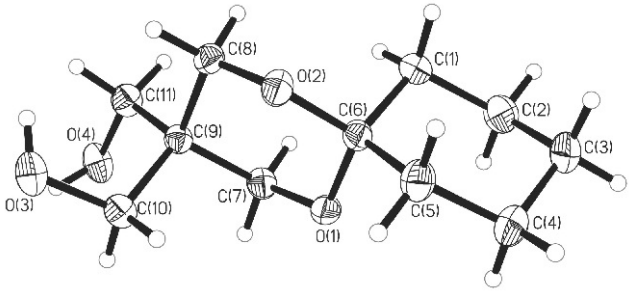
Crystal structure of 3,3-dihydroxymethyl-1,6-dioxa-spiro[5.5]undecane, $C_{11}H_{20}O_4$

Lin Yuan*, Jiang-Hua Shi and Min Zhang

Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou 425100, Hunan, P. R. China

Received March 29, 2011, accepted and available on-line November 16, 2011; CCDC no. 1267/3484



Abstract

$C_{11}H_{20}O_4$, triclinic, $P\bar{1}$ (no. 2), a = 6.014(2) Å, b = 9.566(3) Å, c = 10.135(4) Å, α = 96.669(6)°, β = 94.985(6)°, γ = 97.419(6)°, V = 571.2 Å^3, Z = 2, $R_{gf}(F)$ = 0.053, $wR_{ref}(F^2)$ = 0.179, T = 298 K.

Source of material

Pentaerythritol (3 g, 0.022 mol), cyclohexanone (2 g, 0.02 mol), *N*,*N*-dimethylformamide (40 mL), cyclohexane (15 mL) and *p*-toluene sulfonic acid (0.2 g) were mixed and heated for six hours. The mixture was cooled and then filtered. The solid phase was separated and washed with water and 5% sodium bicarbonate (10 mL). The solvent was evaporated and the product recrystallized from ethylacetate to afford colorless crystals (yield 87 %; m.p. 398 K).

NMR data are available in the CIF file.

Discussion

The title compound was synthesized to be used as a reactive intermediate in organic syntheses [1]. This class of compounds has insecticidal as well as anti-foaming properties [2]. Related structures have been structurally characterized [3,4].

Pentaerythritol is almost insoluble in most organic solvents such as hot benzene. Pentaerythritol monoacetal dissolves in benzene and other organic solvents, and can futher generate a diacetal by condensation with aldehydes. Therefore, the diacetal can be synthesized instead of monoacetal through heterogeneous reaction in organic solvents such as benzene with excess of pentaerythritol. We found that the monoacetal can be synthesized in solvents with strong polarity such as hot dimethylsulfoxide and DMF [5]. The reaction of pentaerythritol with cyclohexanone in a 1:1 molar ratio gave the title compound, which contains two six-membered rings with chair conformation. In the crystal structure, adjacent molecules are connected through O–H···O hydrogen bonding interactions between the oxygen atoms O3 and O4 into a zigzag chain along the *a* axis, forming a herring-bone shape.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.13 × 0.21 × 0.42 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.94 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	54.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4801, 2468
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1749
$N(param)_{refined}$:	138
Programs:	SHELXS-97, SHELXL-97, SHELXTL [6]

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

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	0.2181	0.5567	0.1579	0.072
H(7A)	2*i*	–0.1064	0.8990	0.0802	0.044
H(7B)	2*i*	–0.1017	0.9447	0.2343	0.044
H(8A)	2*i*	0.1606	0.8324	0.3935	0.044
H(8B)	2*i*	0.3062	0.7224	0.3304	0.044
H(4)	2*i*	–0.2472	0.5718	0.0563	0.072
H(11A)	2*i*	–0.0682	0.5851	0.2482	0.049
H(11B)	2*i*	–0.1952	0.7116	0.2971	0.049
H(5A)	2*i*	0.6560	1.1492	0.3132	0.055
H(5B)	2*i*	0.5862	1.0977	0.1610	0.055
H(10A)	2*i*	0.0661	0.7002	–0.0073	0.046
H(10B)	2*i*	0.3011	0.7830	0.0546	0.046
H(1A)	2*i*	0.0898	1.0635	0.4086	0.052
H(1B)	2*i*	0.3328	1.1270	0.4750	0.052
H(2A)	2*i*	0.0664	1.2617	0.2979	0.066
H(2B)	2*i*	0.1265	1.3138	0.4509	0.066
H(3A)	2*i*	0.5047	1.3753	0.4189	0.073
H(3B)	2*i*	0.3555	1.4533	0.3244	0.073
H(4A)	2*i*	0.3823	1.2850	0.1424	0.064
H(4B)	2*i*	0.6274	1.3474	0.2061	0.064

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)	2*i*	0.1740(2)	1.0222(1)	0.1594(1)	0.0435(7)	0.0339(7)	0.0330(6)	0.0021(5)	–0.0041(5)	0.0060(5)
O(2)	2*i*	0.4111(2)	0.9199(1)	0.3041(1)	0.0353(7)	0.0331(7)	0.0471(8)	0.0028(5)	–0.0081(5)	0.0054(5)
O(3)	2*i*	0.2559(2)	0.5828(1)	0.0879(2)	0.0413(8)	0.0385(8)	0.0616(9)	0.0063(6)	0.0082(7)	–0.0048(6)

* Correspondence author (e-mail: zm_zmcm@sina.com)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(9)	2*i*	0.0686(3)	0.7741(2)	0.1918(2)	0.0293(8)	0.0312(9)	0.0317(8)	0.0027(7)	0.0007(7)	0.0027(6)
C(6)	2*i*	0.3306(3)	1.0485(2)	0.2761(2)	0.0356(9)	0.0305(9)	0.0333(9)	0.0040(7)	–0.0024(7)	0.0033(7)
C(7)	2*i*	–0.0117(3)	0.9138(2)	0.1645(2)	0.0332(9)	0.037(1)	0.0381(9)	0.0065(7)	–0.0048(7)	0.0024(7)
C(8)	2*i*	0.2385(3)	0.8069(2)	0.3162(2)	0.041(1)	0.0315(9)	0.0359(9)	0.0034(7)	–0.0041(7)	0.0064(7)
O(4)	2*i*	–0.2933(2)	0.6276(2)	0.1117(2)	0.0312(7)	0.0457(8)	0.0610(9)	0.0013(6)	–0.0019(6)	–0.0113(6)
C(11)	2*i*	–0.1262(3)	0.6690(2)	0.2222(2)	0.0357(9)	0.040(1)	0.044(1)	–0.0008(8)	0.0062(8)	0.0037(8)
C(5)	2*i*	0.5352(3)	1.1422(2)	0.2417(2)	0.041(1)	0.039(1)	0.057(1)	0.0002(8)	0.0068(9)	0.0047(9)
C(10)	2*i*	0.1761(3)	0.7144(2)	0.0706(2)	0.0348(9)	0.040(1)	0.038(1)	0.0022(7)	0.0053(7)	0.0003(7)
C(1)	2*i*	0.2292(3)	1.1207(2)	0.3949(2)	0.055(1)	0.042(1)	0.0335(9)	0.0055(9)	0.0040(8)	0.0028(8)
C(2)	2*i*	0.1825(4)	1.2690(2)	0.3720(2)	0.072(2)	0.046(1)	0.051(1)	0.019(1)	0.017(1)	–0.0010(9)
C(3)	2*i*	0.3926(4)	1.3610(2)	0.3420(2)	0.089(2)	0.032(1)	0.058(1)	0.007(1)	0.001(1)	0.0012(9)
C(4)	2*i*	0.4886(4)	1.2903(2)	0.2210(2)	0.061(1)	0.038(1)	0.060(1)	–0.0043(9)	0.010(1)	0.0096(9)

Acknowledgments. This work was financially supported by Science and Technology Planning Project of Hunan Province (no. 2010NK3007), Science and Technology Planning Project of Yongzhou (no. 2011-6), the Key Project of Hunan University of Science and Engineering (no. 2011) and the Key Subject Construction Project of Hunan Province (no. 2011).

References

1. Marrian, S. F.: The chemical reactions of pentaerythritol and its derivatives. Chem. Rev. **43** (1948) 149-198.
2. Wang, G. W.; Yuan, X. Y.; Liu, Y. C.; Lei, X. G.: Preparation and properties of sulfonate salt-type cleavable surfactants with a 1,3-dioxane ring. J. Am. Oil Chem. Soc. **71** (1994) 727-730.
3. Yuan, X. Y.; Yang, N. F.; Luo, H. A.; Liu, Y. J.: Microwave syntheses of disulfonate salt-type cleavable surfactants with a 1,3-dioxane ring. Chin. J. Org. Chem. **25** (2005) 1049-1052.
4. Zhang, M., Yuan, X. Y.; Ng, S. W.: (5-n-Hexyl-2-hydroxymethyl-1,3-dioxan-2-yl)methanol. Acta Crystallogr. **E66** (2010) o2917.
5. Yuan, X. Y.; Zhang, M.; Ng, S. W.: (5-n-Heptyl-2-hydroxymethyl-1,3-dioxan-2-yl)methanol. Acta Crystallogr. **E66** (2010) o2918.
6. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.