Crystal structure of (1*R*,3*S*,5*R*)-3-hydroxy-2-methylene-6-oxa-bicyclo[3.2.1]octan-7-one, C₈H₁₀O₃

K. Peters^{*,I}, E.-M. Peters^I and T. Linker^{II}^I Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, 70506 Stuttgart, Germany^{II} Universität Potsdam, Institut für Chemie, Karl-Liebknecht-Straße 24–25, 14476 Golm, Germany

Received July 21, 2004, accepted and available on-line November 16, 2004; CCDC no. 1267/1371

Abstract

C₈H₁₀O₃, orthorhombic, *Pbca* (no. 61), *a* = 10.592(3) Å, *b* = 11.877(3) Å, *c* = 36.833(9) Å, *V* = 4633.6 Å³, *Z* = 24, *R*_{gt}(*F*) = 0.091, *wR*_{ref}(*F*²) = 0.219, *T* = 293 K.

Source of material

The title compound was prepared by photooxygenation of the corresponding methyl-substituted double bond and subsequent reduction [1].

Discussion

Three molecules of the title compound form the asymmetric unit. These molecules are connected by the intermolecular hydrogen bridges O11...H31 (180 pm) and O21...H11 (166 pm). Via the intermolecular hydrogen bridge O31...H21 (168 pm) these units form a zig-zag chain along [010].

The large *R* values are caused by the poor crystal quality, which probably results from the pseudosymmetry or the strong anisotropic intermolecular bonding leading to needle-like individuals.

Table 1. Data collection and handling.

Crystal:	colorless lath, size 0.07 × 0.2 × 0.85 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
<i>μ</i> :	1.01 cm ^{−1}
Diffraction, scan mode:	Bruker AXS P4, <i>ω</i>
2 θ _{max} :	54.98°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	6612, 5320
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 1726
<i>N</i> (<i>param</i>) _{refined} :	298
Programs:	SHELXS-97 [2], SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(11)	8c	0.6498	0.2167	0.1208	0.08
H(11A)	8c	0.6137	0.2119	0.1761	0.08
H(13)	8c	0.8558	0.2204	0.2458	0.08
H(14A)	8c	0.7954	0.0379	0.2641	0.08
H(14B)	8c	0.8484	0.0180	0.2243	0.08
H(15)	8c	0.6224	−0.0407	0.2340	0.08
H(17A)	8c	0.5553	0.0295	0.1768	0.08
H(17B)	8c	0.6945	−0.0139	0.1727	0.08
H(18A)	8c	0.8948	0.2592	0.1516	0.08
H(18B)	8c	0.9593	0.2729	0.1909	0.08
H(21)	8c	0.5689	0.4088	0.1067	0.08
H(21A)	8c	0.3903	0.3764	0.0934	0.08
H(23)	8c	0.2193	0.3383	0.1797	0.08
H(24A)	8c	0.3100	0.1471	0.1632	0.08
H(24B)	8c	0.1607	0.1553	0.1608	0.08
H(25)	8c	0.2178	0.1063	0.1017	0.08
H(27A)	8c	0.3821	0.2003	0.0717	0.08
H(27B)	8c	0.4365	0.1473	0.1074	0.08
H(28A)	8c	0.5318	0.4271	0.1653	0.08
H(28B)	8c	0.4186	0.4191	0.1944	0.08
H(31)	8c	0.7840	0.0619	0.1030	0.08
H(31A)	8c	0.8995	0.0078	0.0421	0.08
H(33)	8c	0.6066	0.0822	−0.0031	0.08
H(34A)	8c	0.6865	0.2646	−0.0118	0.08
H(34B)	8c	0.6750	0.2562	0.0311	0.08
H(35)	8c	0.8919	0.2940	0.0077	0.08
H(37A)	8c	0.8653	0.2276	0.0679	0.08
H(37B)	8c	0.9875	0.1768	0.0503	0.08
H(38A)	8c	0.6425	−0.0361	0.0844	0.08
H(38B)	8c	0.5438	−0.0122	0.0520	0.08

* Correspondence author (e-mail: karlpeters@gmx.net)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
O(11)	8c	0.7037(3)	0.1578(3)	0.13316(8)	0.077(3)	0.087(3)	0.039(2)	0.035(2)	−0.008(2)	−0.002(2)
C(11)	8c	0.6833(5)	0.1597(4)	0.1713(1)	0.048(3)	0.065(4)	0.043(3)	0.016(3)	−0.008(2)	−0.006(3)
C(12)	8c	0.7963(5)	0.2016(4)	0.1913(1)	0.044(3)	0.048(3)	0.046(3)	0.002(3)	0.009(2)	0.006(2)
C(13)	8c	0.7881(5)	0.1827(5)	0.2324(1)	0.065(4)	0.070(4)	0.047(3)	0.002(3)	−0.003(3)	−0.001(3)
C(14)	8c	0.7851(5)	0.0569(5)	0.2386(1)	0.079(4)	0.072(4)	0.059(3)	0.009(4)	−0.004(3)	0.016(3)
C(15)	8c	0.6528(6)	0.0325(5)	0.2253(2)	0.103(5)	0.057(4)	0.074(4)	−0.009(4)	0.011(4)	0.002(3)
O(15)	8c	0.5802(4)	0.1260(3)	0.2411(1)	0.078(3)	0.073(3)	0.082(3)	−0.006(3)	0.026(2)	−0.004(2)
C(16)	8c	0.6568(6)	0.2177(6)	0.2450(1)	0.071(4)	0.075(4)	0.038(3)	−0.001(4)	0.004(3)	0.010(3)
O(16)	8c	0.6171(4)	0.3043(3)	0.25623(9)	0.123(4)	0.059(2)	0.066(2)	0.009(3)	0.023(2)	−0.013(2)
C(17)	8c	0.6420(5)	0.0428(4)	0.1841(1)	0.069(4)	0.063(4)	0.073(4)	−0.006(3)	0.006(3)	−0.009(3)
C(18)	8c	0.8922(5)	0.2489(5)	0.1766(2)	0.063(4)	0.093(5)	0.065(3)	0.001(4)	0.003(3)	−0.002(3)
O(21)	8c	0.5598(3)	0.3215(3)	0.10640(9)	0.054(2)	0.054(2)	0.085(2)	0.007(2)	0.007(2)	0.016(2)
C(21)	8c	0.4254(4)	0.3203(4)	0.1101(1)	0.044(3)	0.044(3)	0.046(3)	−0.002(3)	−0.003(2)	0.004(2)
C(22)	8c	0.3820(4)	0.3490(4)	0.1477(1)	0.051(3)	0.039(3)	0.038(2)	0.000(3)	−0.008(2)	0.002(2)
C(23)	8c	0.2478(5)	0.3129(4)	0.1558(1)	0.059(4)	0.050(3)	0.051(3)	0.002(3)	0.013(3)	0.008(3)
C(24)	8c	0.2398(5)	0.1852(4)	0.1516(1)	0.057(4)	0.050(3)	0.092(4)	−0.006(3)	0.015(3)	0.014(3)
C(25)	8c	0.2475(5)	0.1792(4)	0.1108(2)	0.064(4)	0.036(3)	0.087(4)	−0.008(3)	−0.017(3)	−0.009(3)
O(25)	8c	0.1635(3)	0.2716(3)	0.0993(1)	0.059(3)	0.069(3)	0.098(3)	−0.002(2)	−0.035(2)	0.003(2)
C(26)	8c	0.1614(5)	0.3511(5)	0.1259(2)	0.047(4)	0.047(4)	0.098(5)	−0.007(3)	−0.002(3)	0.013(3)
O(26)	8c	0.1020(4)	0.4349(3)	0.1226(1)	0.061(3)	0.060(3)	0.144(4)	0.020(2)	0.000(3)	0.017(3)
C(27)	8c	0.3796(5)	0.2042(4)	0.0980(1)	0.061(4)	0.048(3)	0.050(3)	0.002(3)	0.000(3)	−0.012(2)
C(28)	8c	0.4508(6)	0.4037(5)	0.1714(1)	0.085(5)	0.085(4)	0.069(4)	0.002(4)	−0.017(3)	−0.020(3)
O(31)	8c	0.8638(4)	0.0297(3)	0.09331(8)	0.101(3)	0.093(3)	0.043(2)	0.058(3)	0.007(2)	0.014(2)
C(31)	8c	0.8471(5)	0.0594(4)	0.0564(1)	0.054(3)	0.060(3)	0.039(3)	0.020(3)	0.008(2)	0.007(2)
C(32)	8c	0.7131(5)	0.0449(4)	0.0432(1)	0.059(3)	0.038(3)	0.054(3)	−0.007(3)	0.015(3)	−0.011(2)
C(33)	8c	0.6907(5)	0.0992(4)	0.0066(1)	0.052(4)	0.068(4)	0.063(3)	0.001(3)	−0.013(3)	−0.013(3)
C(34)	8c	0.7142(6)	0.2241(5)	0.0097(2)	0.098(5)	0.065(4)	0.061(3)	0.026(4)	−0.018(3)	0.008(3)
C(35)	8c	0.8549(6)	0.2200(4)	0.0127(1)	0.085(5)	0.048(3)	0.060(3)	−0.015(3)	−0.004(3)	0.007(3)
O(35)	8c	0.8912(4)	0.1380(3)	−0.01535(9)	0.093(3)	0.077(3)	0.046(2)	−0.001(3)	0.015(2)	0.005(2)
C(36)	8c	0.7945(6)	0.0647(5)	−0.0191(1)	0.091(5)	0.063(4)	0.039(3)	0.008(4)	−0.004(3)	0.007(3)
O(36)	8c	0.8021(5)	−0.0130(3)	−0.0396(1)	0.163(5)	0.084(3)	0.048(2)	0.008(3)	0.005(2)	−0.023(2)
C(37)	8c	0.8960(5)	0.1772(4)	0.0492(1)	0.050(3)	0.076(4)	0.060(3)	−0.008(3)	−0.003(3)	−0.004(3)
C(38)	8c	0.6247(6)	−0.0060(5)	0.0616(2)	0.094(5)	0.091(5)	0.079(4)	−0.040(4)	0.032(4)	−0.029(4)

References

1. Fröhlich, L.; Linker, T.: Simple and Diastereoselective Synthesis of an A-Ring Precursor of Dihydroxyvitamin D₃ (Calcitriol) by Photooxygenation. *Synlett* **13** (2004) 2725–2727.
2. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
3. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.