Crystal structure of 2,3-*O*-dimethyl-5,6-*O*-isopropylidene-L-ascorbic acid, C₁₁H₁₆O₆

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Abstract

C₁₁H₁₆O₆, trigonal, *P*3₂ (No. 145), *a* = 12.043(2) Å, *c* = 7.666(2) Å, *V* = 962.9 Å³, *Z* = 3, *R*_{gt}(*F*) = 0.037, *wR*_{ref}(*F*²) = 0.122, *T* = 293 K.

Source of material

2,3-*O*-Dimethyl-5,6-*O*-isopropylidene-L-ascorbic acid was synthesized as described elsewhere [1]. Crystals were grown by slow evaporation of a solution of the title compound in isopropanol/water (1:1).

Experimental details

All hydrogen atoms were placed at calculated sites using a riding model. Isotropic displacement parameters were used which were fixed to a value corresponding to the *U*_{eq} value of the bonded atom multiplied with a factor of 1.2. In the figure, the displacement ellipsoids are drawn at the 50% probability level. The hydrogen atoms are shown in arbitrary size.

Discussion

Due to the well established importance in food science and nutrition [2], the chemistry of L-ascorbic acid has intensively been studied [1]. The four hydroxy groups of this compound allow selective alkylation, and a variety of such chiral derivatives have been prepared. However, only a few crystal structures of such compounds have been reported [3–6]. In the title compound, a vitamin C derivative, the four hydroxy groups of L-ascorbic acid are all alkylated. The molecule contains two five membered rings with different conformations. The furanone ring is practically planar: the deviation of the mean plane is less than 0.04 Å for the five ring atoms, whereas the 1,3-dioxolane ring has a puckered conformation corresponding to an envelope.

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.2 × 0.3 × 0.4 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	1.38 cm ⁻¹
Diffractometer, scan mode:	Stoe IPDS, φ
2θ _{max} :	48.2°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	6056, 1897
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 1683
<i>N</i> (<i>param</i>) _{refined} :	156
Programs:	SHELXS-97 [7], SHELXL-97 [8]

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(4)	3a	0.4836	0.1562	−0.0775	0.061
H(21A)	3a	0.1009	−0.0762	0.4759	0.109
H(21B)	3a	0.2481	0.0224	0.4886	0.109
H(21C)	3a	0.1984	−0.0960	0.3636	0.109
H(5)	3a	0.3877	0.2242	−0.2919	0.063
H(6A)	3a	0.6270	0.3708	−0.2006	0.077
H(6B)	3a	0.5663	0.4034	−0.3614	0.077
H(41A)	3a	0.0625	−0.1474	−0.1702	0.121
H(41B)	3a	0.0548	−0.0487	−0.0445	0.121
H(41C)	3a	0.1005	−0.1413	0.0266	0.121
H(56A)	3a	0.3401	0.5156	0.0363	0.201
H(56B)	3a	0.4867	0.6175	0.0325	0.201
H(56C)	3a	0.4392	0.4851	0.1247	0.201
H(56D)	3a	0.3234	0.5052	−0.3028	0.211
H(56E)	3a	0.4189	0.4770	−0.4062	0.211
H(56F)	3a	0.4682	0.6112	−0.3182	0.211

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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(3)	3a	0.4815(2)	0.2681(2)	0.1133(2)	0.0436(9)	0.063(1)	0.052(1)	0.0207(8)	−0.0072(8)	0.0000(8)
O(2)	3a	0.1683(2)	0.0404(2)	0.2732(3)	0.048(1)	0.080(1)	0.055(1)	0.0326(9)	0.0105(8)	0.0162(9)
O(5)	3a	0.3571(2)	0.3356(2)	−0.1334(3)	0.0464(9)	0.055(1)	0.083(2)	0.0229(8)	−0.0069(9)	0.0075(9)
O(6)	3a	0.5668(2)	0.4927(2)	−0.1436(4)	0.053(1)	0.056(1)	0.114(2)	0.0158(8)	−0.002(1)	−0.004(1)
O(4)	3a	0.2326(2)	0.0021(2)	−0.1143(3)	0.068(1)	0.054(1)	0.061(1)	0.0110(9)	0.003(1)	−0.0079(9)
C(2)	3a	0.2766(2)	0.1058(2)	0.1739(4)	0.042(1)	0.057(1)	0.051(2)	0.026(1)	0.003(1)	0.010(1)
O(1)	3a	0.4042(2)	0.2773(2)	0.3752(3)	0.082(1)	0.090(2)	0.049(2)	0.039(1)	−0.012(1)	−0.013(1)
C(3)	3a	0.2995(2)	0.0875(2)	0.0090(3)	0.049(1)	0.048(1)	0.044(2)	0.023(1)	0.001(1)	0.003(1)
C(4)	3a	0.4303(2)	0.1930(2)	−0.0430(4)	0.045(1)	0.051(1)	0.056(2)	0.023(1)	0.003(1)	−0.004(1)
C(21)	3a	0.1799(3)	−0.0335(3)	0.4118(5)	0.064(2)	0.080(2)	0.071(2)	0.033(2)	0.015(1)	0.028(2)
C(5)	3a	0.4282(2)	0.2769(2)	−0.1883(4)	0.050(1)	0.052(1)	0.046(2)	0.019(1)	0.003(1)	−0.001(1)
C(1)	3a	0.3876(2)	0.2224(3)	0.2380(4)	0.051(1)	0.062(2)	0.042(2)	0.028(1)	−0.008(1)	0.001(1)
C(6)	3a	0.5596(3)	0.3877(3)	−0.2367(4)	0.061(2)	0.054(2)	0.064(2)	0.017(1)	0.009(1)	0.006(1)
C(56)	3a	0.4390(3)	0.4715(3)	−0.1416(6)	0.066(2)	0.052(2)	0.128(3)	0.028(1)	−0.010(2)	0.002(2)
C(41)	3a	0.1016(3)	−0.0917(3)	−0.0721(5)	0.066(2)	0.066(2)	0.073(3)	0.006(2)	−0.008(2)	0.000(2)
C(561)	3a	0.4250(4)	0.5276(5)	0.029(1)	0.091(3)	0.098(3)	0.219(8)	0.051(3)	−0.007(3)	−0.061(4)
C(562)	3a	0.4097(5)	0.5207(5)	−0.307(1)	0.119(4)	0.088(3)	0.210(7)	0.048(3)	−0.028(4)	0.052(4)

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