Crystal structure of 4-hydroxy-6-propenyl-5,6-dihydro-pyran-2-one, $C_8H_{10}O_3$

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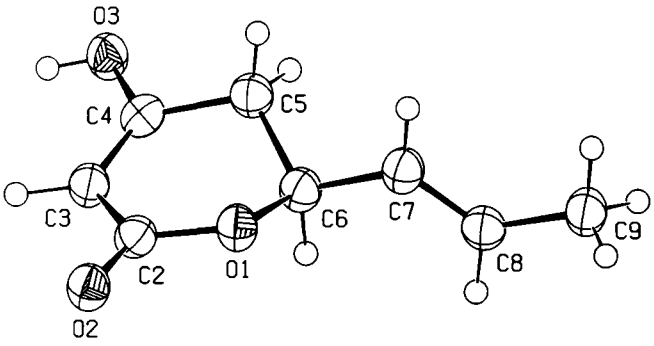

Abstract

$C_8H_{10}O_3$, monoclinic, $P12_1/c1$ (no. 14), $a = 5.1640(2)$ Å, $b = 19.979(1)$ Å, $c = 7.5840(4)$ Å, $\beta = 104.540(2)°$, $V = 757.4$ Å^3, $Z = 4$, $R_{gt}(F) = 0.049$, $wR_{ref}(F^2) = 0.169$, $T = 293$ K.

Source of material

The title compound was synthesized, in 80 % overall yield, by the reaction in tetrahydrofuran, of ethyl acetoacetate dianion with crotonaldehyde, followed by ester hydrolysis in basic medium (NaOH, 0.5 M) and lactonization under acidic conditions (HCl, 36 %) [1]. It was purified by crystallization from chloroform at 25 °C.

Discussion

Dihydropyran-2,4-diones are known to have biological activity [2-4]. The title compound has shown significant activities as antioxidant ($EC_{50} = 1.44$ mM) [5] and against *Biomphalaria glabrata* egg masses ($LC_{90} = 40.26$ ppm) [1].

The conformation of the pyranic ring is of the half boat as indicated by the Cremer-Pople puckering parameters [6] being $q_2 = 0.39$ Å, $q_3 = 0.21$ Å, $\theta = 288°$, $\varphi = 62.0°$, $Q = 0.44$ Å. Calculating the mean least-squares plane through of the atoms C6, C7, C8, and C9, we observe co-planarity. The distances of these atoms to the average reference plane are 0.0071(1) Å, 0.010(2) Å, 0.013(2) Å, and 0.0052(3) Å, respectively. The dihedral angle between this plane and the mean plane passing through atoms O1, C2, C3, C4, and C5 of the heterocyclic ring is 83.1°. The molecules in the packing form ribbons along [201] parallel to the $(10\bar{2})$ plane that are held together by one typical hydrogen bond O3–H···O2^i, where d(O3—H) = 0.82 Å, d(H···O2^i) = 1.874 Å, d(O3···O2^i) = 2.602(2) Å, ∠O3–H···O2^i = 147°, symmetry code (i): $x+1,-y-½,z+½$. Within the experimental accuracy, bond lengths and angles are in good agreement with the values found in the literature.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.25 × 0.3 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.03 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius KappaCCD, ω
$2\theta_{max}$:	49.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2515, 1325
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1021
$N(param)_{refined}$:	100
Programs:	SHELXS-97 [7], SHELXL-97 [8], ORTEP-3 [9], WinGX [10]

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

Atom	Site	x	y	z	U_{iso}
H(3)	4e	1.6129	–0.1664	1.8952	0.055
H(5A)	4e	1.1607	–0.0662	1.6637	0.042
H(5B)	4e	0.9614	–0.1080	1.7610	0.042
H(7)	4e	0.5493	–0.0899	1.5130	0.042
H(8)	4e	0.6862	–0.0795	1.1613	0.044
H(3')	4e	1.3877	–0.2636	1.7487	0.040
H(9A)	4e	0.3477	0.0109	1.1467	0.048
H(9B)	4e	0.2106	–0.0257	1.2802	0.048
H(9C)	4e	0.2086	–0.0507	1.1044	0.048
H(6)	4e	1.0119	–0.1366	1.3791	0.040

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)	4e	0.8186(3)	–0.20659(7)	1.4972(2)	0.0325(9)	0.0302(9)	0.0374(9)	–0.0006(6)	0.0026(6)	0.0021(6)
O(2)	4e	0.9408(3)	–0.31120(8)	1.5556(2)	0.0371(9)	0.0313(9)	0.0382(9)	–0.0030(6)	0.0044(7)	0.0011(6)
C(2)	4e	1.0050(4)	–0.2520(1)	1.5800(3)	0.035(1)	0.034(1)	0.030(1)	0.0001(9)	0.0074(9)	0.0015(8)
O(3)	4e	1.5020(3)	–0.13717(7)	1.8558(2)	0.0347(9)	0.0320(9)	0.0389(9)	0.0015(6)	0.0003(7)	–0.0015(7)
C(5)	4e	1.0753(4)	–0.1143(1)	1.6683(3)	0.035(1)	0.032(1)	0.036(1)	–0.0004(9)	0.0059(9)	–0.0013(9)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(7)	4*e*	0.6538(4)	–0.0970(1)	1.4198(3)	0.034(1)	0.035(1)	0.036(1)	0.0000(9)	0.005(1)	0.0018(9)
C(4)	4*e*	1.2900(4)	–0.1643(1)	1.7408(3)	0.032(1)	0.037(1)	0.029(1)	–0.0028(9)	0.0058(9)	0.0007(9)
C(8)	4*e*	0.5727(4)	–0.0713(1)	1.2546(3)	0.039(1)	0.032(1)	0.036(1)	–0.0007(9)	0.004(1)	–0.0029(9)
C(3)	4*e*	1.2574(4)	–0.2292(1)	1.6934(3)	0.033(1)	0.032(1)	0.033(1)	0.0007(9)	0.0054(9)	0.0017(9)
C(9)	4*e*	0.3200(4)	–0.0330(1)	1.1852(3)	0.041(1)	0.035(1)	0.039(1)	0.003(1)	0.001(1)	0.002(1)
C(6)	4*e*	0.9029(4)	–0.1368(1)	1.4847(3)	0.036(1)	0.029(1)	0.034(1)	–0.0021(9)	0.0048(9)	0.0004(9)

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