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Redetermination of 3-[(*Z*)-1-hydroxy-3-oxobutenyl]-2*H*-chromen-2-one at 193 K

Xiangshan Wang,[a,b]* Zhaosen Zeng,[a] Yuling Li[a] and Daqing Shi[a]

[a]Department of Chemistry, Xuzhou Normal University, Xuzhou 221116, People's Republic of China, and [b]The Key Laboratory of Biotechnology of Medical Plants of Jiangsu Province, Xuzhou 221116, People's Republic of China

Correspondence e-mail:
xswang1974@yahoo.com

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The structure of the title compound, $C_{13}H_{10}O_4$, which was synthesized by the reaction of salicylaldehyde and 4-hydroxy-6-methyl-2*H*-pyran-2-one in the presence of triethylbenzylammonium chloride in aqueous media, was previously determined at room temperature [March, Moreno-Manas, Roca, Germain, Piniella & Dideberg (1986). *J. Heterocycl. Chem*. **23**, 1511–1153]. As in the present determination, the X-ray analysis revealed that the title compound is in the enol form, which was confirmed by ^{1}H NMR data. Weak intermolecular C—H···O interactions connect molecules into a two-dimensional framework.

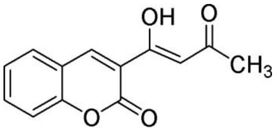
Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.058
wR factor = 0.170
Data-to-parameter ratio = 12.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Coumarins have attracted intense interest in recent years because of their diverse pharmacological properties; some of these compounds possess anti-estrogenic and anti-ovulatory activity (Agrawal *et al.*, 1978), antibacterial activity (Desai & Mehta, 1997; Miky *et al.*, 1997), anti-oxidant activity (Vladimirov *et al.*, 1991), antimicrobial activity (Rao *et al.*, 1982; 1983) and anti-inflammatory activity (Kulkarni *et al.*, 1981). The structure of the title compound, (I), has already been determined at room temperature (March *et al.*, 1986), but the H atom of the enol hydroxyl group was not located. In order to confirm the predicted structure, the X-ray analysis of (I) was repeated at low temperature.



(I)

In the title compound, the C10—C11 bond distance of 1.372 (3) Å is statistically equivalent to the value of 1.368 (3) Å in the original determination (March *et al.*, 1986),

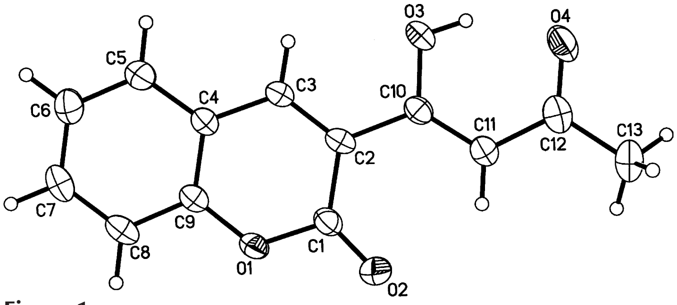


Figure 1
The molecular structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

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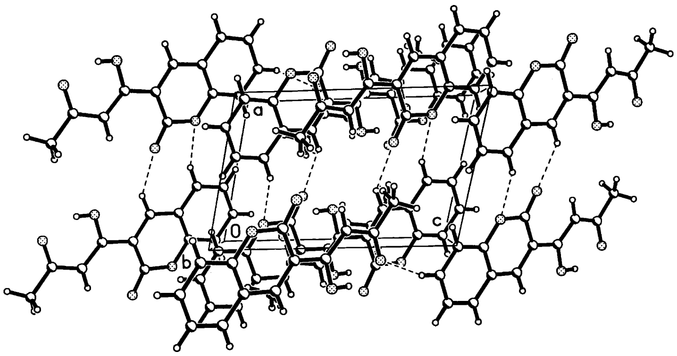

Figure 2
The molecular packing of (I). Dashed lines indicate hydrogen bonds.

indicating double-bond character in both cases. In addition, the longer bond, 1.325 (2) Å, for C10—O3 compared to 1.257 (3) Å for C12—O4 coupled with the ^{1}H NMR data (see *Experimental*) confirm that the title structure is in the enol form. The atoms of the pyran ring (C1–C4/C9/O1) are essentially coplanar (Fig. 1), with a maximun deviation of 0.018 (2) Å for O1; this ring forms a dihedral angle of 1.4 (1)° with the benzene ring (C4–C9).

In addition to an intramolecular O—H···O hydrogen bond, weak C—H···O interactions connect molecules into a two-dimensional framework (Table 2 and Fig. 2).

Experimental

The title compound, (I), was prepared by the reaction of salicylaldehyde (0.25 g, 2 mmol) and 4-hydroxy-6-methyl-2*H*-pyran-2-one (0.25 g, 2 mmol) in the presence of triethylbenzylammonium chloride (0.1 g) in water at 363 K for 8 h (yield 93.5%, m.p. 421–423 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. Elemental analysis calculated: C 67. 82, H 4.38%; found: C 67.71, H 4.45%. ^{1}H NMR (CDCl$_3$): δ 2.37 (*s*, 3H, CH$_3$), 7.06 (*s*, 1H, CH═), 7.35–7.72 (*m*, 4H, ArH), 8.68 (*s*, 1H, CH═), 15.89 (*s*, 1H, OH); IR (cm^{-1}): 3255 (*b*, OH), 3059 (Ar—H), 2939 (C—H), 1737 (C═O), 1580, 1474 (benzene ring).

Crystal data

$C_{13}H_{10}O_4$
M_r = 230.21
Triclinic, $P\overline{1}$
a = 6.7577 (19) Å
b = 8.799 (3) Å
c = 9.890 (4) Å
α = 83.17 (4)°
β = 76.17 (3)°
γ = 71.10 (3)°
V = 539.7 (3) Å^3

Z = 2
D_x = 1.417 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 1738 reflections
θ = 3.2–25.3°
μ = 0.11 mm^{-1}
T = 193 (2) K
Block, light yellow
0.41 × 0.37 × 0.15 mm

Data collection

Rigaku Mercury diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{\min}$ = 0.958, $T_{\max}$ = 0.984
5300 measured reflections
1958 independent reflections

1461 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.024
$\theta_{\max}$ = 25.4°
$h = -7 \rightarrow 8$
$k = -10 \rightarrow 9$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.170
S = 1.08
1958 reflections
157 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0949P)^2 + 0.0935P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.39 e Å^{-3}
$\Delta\rho_{\min}$ = −0.23 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.372 (2)	C1—C2	1.466 (3)
O1—C1	1.379 (2)	C2—C3	1.357 (3)
O2—C1	1.206 (2)	C2—C10	1.476 (3)
O3—C10	1.325 (2)	C10—C11	1.372 (3)
O4—C12	1.257 (3)	C11—C12	1.430 (3)
C9—O1—C1	123.13 (15)	C2—C3—C4	121.80 (18)
O2—C1—O1	115.24 (17)	O3—C10—C2	113.41 (17)
O2—C1—C2	127.74 (18)	C11—C10—C2	125.88 (18)
O1—C1—C2	117.02 (17)	C10—C11—C12	120.6 (2)
C3—C2—C1	119.56 (18)	O4—C12—C11	121.3 (2)
C3—C2—C10	119.95 (17)	O4—C12—C13	119.4 (2)
C1—C2—C10	120.49 (17)	C11—C12—C13	119.3 (2)
C9—O1—C1—O2	179.25 (16)	C2—C3—C4—C9	−0.9 (3)
C9—O1—C1—C2	−0.6 (3)	C1—O1—C9—C4	0.9 (3)
C1—C2—C3—C4	1.2 (3)	C3—C4—C9—O1	−0.1 (3)
C10—C2—C3—C4	−179.02 (15)	C1—C2—C10—C11	0.4 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O4	0.84	1.74	2.493 (2)	148
C3—H3*A*···O2$^{\rm i}$	0.95	2.52	3.358 (2)	147
C5—H5···O1$^{\rm i}$	0.95	2.53	3.414 (3)	155
C7—H7···O4$^{\rm ii}$	0.95	2.45	3.371 (3)	163

Symmetry codes: (i) $x-1, y, z$; (ii) $x, y+1, z-1$.

The H atoms were positioned geometrically and refined as riding, with C—H = 0.95–0.98 Å and O—H = 0.84 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,O) or $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl.

Data collection: *CRYSTALCLEAR* (Rigaku, 1999); cell refinement: *CRYSTALCLEAR*; data reduction: *CrystalStructure* (Rigaku/MSC 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXL97*.

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