Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Redetermination of 7-hydroxy-4-methyl-2*H*-1-benzopyran-2-one

Shu-Ping Yang,[a]* Li-Jun Han,[b] Da-Qi Wang[c] and Hai-Tao Xia[a]

[a]Department of Chemical Engineering, Huaihai Institute of Technology, Lianyungang 222005, People's Republic of China, [b]Department of Mathematics and Science, Huaihai Institute of Technology, Lianyungang 222005, People's Republic of China, and [c]College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China
Correspondence e-mail: yangshuping@hhit.edu.cn

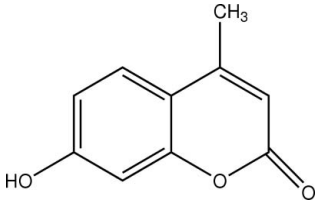
Received 12 October 2007; accepted 5 November 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.098; data-to-parameter ratio = 7.3.

The crystal structure determination of the title compound (also known as 7-hydroxy-4-methylcoumarin), $C_{10}H_8O_3$, has been reported previously [Shimizu, Kashino & Haisa (1975). *Acta Cryst.* B**31**, 1287–1292], but with incomplete crystallographic data. The present redetermination confirms the previous study, but with higher precision and full crystallographic data.

Related literature

For the previously determined crystal structure, see: Shimizu *et al.* (1975). For related literature regarding the uses of 7-hydroxycoumarin derivatives, see: Brühlmann *et al.* (2001); Rollinger *et al.* (2004); Sharma *et al.* (2005). For hydrogen bonding motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{10}H_8O_3$
M_r = 176.16
Orthorhombic, $P2_12_12_1$
a = 5.229 (3) Å
b = 11.875 (6) Å
c = 13.161 (6) Å
V = 817.3 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.48 × 0.40 × 0.31 mm

Data collection

Siemens SMART 1000 CCD area detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.951, T_{max} = 0.968
4236 measured reflections
869 independent reflections
732 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.098
S = 1.08
869 reflections
119 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2^{i}	0.82	1.89	2.712 (3)	175
C2—H2···O2^{ii}	0.93	2.51	3.410 (4)	162

Symmetry codes: (i) $-x-\frac{1}{2}, -y, z+\frac{1}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

We acknowledge the financial support of the Huaihai Institute of Technology Science Foundation.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2107).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Brühlmann, C., Ooms, F., Carrupt, P.-A., Testa, B., Catto, M., Leonetti, F., Altomare, C. & Carotti, A. (2001). *J. Med. Chem.* **44**, 3195–3198.
Rollinger, J. M., Hornick, A., Langer, T., Stuppner, H. & Prast, H. (2004). *J. Med. Chem.* **47**, 6248–6254.
Sharma, S. D., Rajor, H. K., Chopra, S. & Sharma, R. K. (2005). *Biometals*, **18**, 143–154.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Shimizu, S., Kashino, S. & Haisa, M. (1975). *Acta Cryst.* B**31**, 1287–1292.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-Ray Instruments Inc., Madison, Wisconsin, USA.

 doi:10.1107/S1600536807056085

supplementary materials

Acta Cryst. (2007). E**63**, o4643 [**doi:10.1107/S1600536807056085**]

Redetermination of 7-hydroxy-4-methyl-2*H*-1-benzopyran-2-one

S.-P. Yang, L.-J. Han, D.-Q. Wang and H.-T. Xia

Comment

7-Hydroxycoumarin derivatives have a broad range of biological activities, including as antioxidants (Sharma *et al.*, 2005), and for the inhibition of acetylcholinesterase and monoamine oxidase (Rollinger *et al.*,2004; Brühlmann *et al.*, 2001). The crystal structure of compound (I) has been reported previously, but with incomplete crystallographic data (Shimizu *et al.*,1975). We redetermined the crystal structure of compound (I), which we describe in this paper.

In the molecule of (I), the average C—C and C—O distances are 1.392 (4)Å and 1.371 (2) Å, respectively, slightly less than the previously reported values [1.40Å and 1.373 Å; Shimizu *et al.* (1975)] (Fig. 1).

In the crystal packing of (I), the molecules are linked into a $C_2^2(16)$ chain by O—H···O hydrogen bonds (Bernstein *et al.*, 1995) along the [0 0 1] direction (Fig. 2 and Table 1), with atom O3 acting as hydrogen-bond donor to atom O2 in the molecule at $(-1/2-x,-y,1/2+z)$. The molecules are further linked by C—H···O hydrogen bonds, between atom C2 and atom O2 at $(1/2+x,1/2-y,-z)$, forming a $C_2^2(8)$ chain along the [1 0 0] direction (Fig. 3 and Table 1),.

Experimental

The reaction mixture containing resorcinol (1.10 g, 10 mmol), acetoacetic ester (1.3 ml, 10 mmol) and phosphoric acid (5.3 ml) was stirred at 343–353 K for 12 h, and then poured into water. The solid obtained was filtered off, washed with water and dried at room temperature. Colourless crystals of (I) suitable for X-ray structure analysis were obtained by slow evaporation of a solution of the crude product in acetone-petroleum benzine (1:1) over two weeks. (m. p. 463–465 K).

Refinement

All H atoms were positioned geometrically and refined as riding on their parent atoms, with C—H = 0.96 Å, O—H = 0.82 Å, and U_{iso}(H)= 1.5U_{eq}(C,*O*) for methyl H atoms and hydroxy H atoms, and C—H = 0.93Å and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms. In the absence of significant anomalous scattering effects Friedel pairs have been merged.

Figures

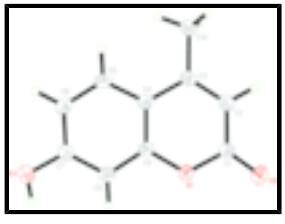


Fig. 1. The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

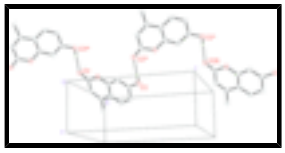


Fig. 2. Part of the crystal structure of (I), viewed down the *b* axis, showing the formation of a [0 0 1] $C_2^2(16)$ chain built from O—H···O hydrogen bonds. For clarity, H atoms not involved in the motifs shown have been omitted. Dashed lines indicate hydrogen bonds [symmetry codes: (*)-1/2 − *x*,-*y*,1/2 + *z*; (#)-1/2 − *x*,-*y*,-1/2 + *z*; (&)*x*,*y*,1 + *z*].

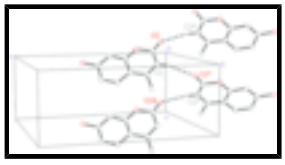


Fig. 3. Part of the crystal structure of (I), viewed down the *b* axis, showing the formation of a [1 0 0] $C_2^2(8)$ chain built from C—H···O hydrogen bonds. For clarity, H atoms not involved in the motifs shown have been omitted. Dashed lines indicate hydrogen bonds [symmetry codes: (*)1/2 + *x*,1/2 − *y*,-*z*; (#)-1/2 + *x*,1/2 − *y*,-*z*; (&)1 + *x*,*y*,*z*].

7-hydroxy-4-methyl-2*H*-1-Benzopyran-2-one

Crystal data

$C_{10}H_8O_3$	D_x = 1.432 Mg m^{-3}
M_r = 176.16	Melting point: 463 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 1640 reflections
a = 5.229 (3) Å	θ = 2.3–25.2º
b = 11.875 (6) Å	μ = 0.11 mm^{-1}
c = 13.161 (6) Å	*T* = 298 (2) K
V = 817.3 (7) Å^3	Columnar, colourless
Z = 4	0.48 × 0.40 × 0.31 mm
F_{000} = 368	

Data collection

Siemens SMART 1000 CCD area detector diffractometer	869 independent reflections
Radiation source: fine-focus sealed tube	732 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.044
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.3º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −6→5
T_{min} = 0.951, T_{max} = 0.968	*k* = −14→12
4236 measured reflections	*l* = −14→15

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.1639P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.098	$(\Delta/\sigma)_{max} < 0.001$

S = 1.08	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
869 reflections	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
119 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 565 Freidel pairs
Secondary atom site location: difference Fourier map	Flack parameter: ?

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.0817 (4)	0.08023 (15)	0.22571 (13)	0.0425 (5)
O2	−0.1981 (4)	0.10501 (18)	0.06697 (14)	0.0529 (6)
O3	0.1286 (5)	0.02230 (17)	0.57207 (14)	0.0511 (6)
H3	0.0029	−0.0188	0.5685	0.077*
C1	−0.0515 (6)	0.1327 (2)	0.1341 (2)	0.0391 (7)
C2	0.1438 (6)	0.2161 (2)	0.1257 (2)	0.0416 (7)
H2	0.1637	0.2535	0.0641	0.050*
C3	0.3006 (6)	0.2430 (2)	0.2032 (2)	0.0365 (7)
C4	0.2715 (5)	0.1833 (2)	0.2979 (2)	0.0349 (7)
C5	0.4233 (6)	0.1998 (2)	0.3841 (2)	0.0399 (7)
H5	0.5588	0.2504	0.3809	0.048*
C6	0.3779 (6)	0.1437 (2)	0.4730 (2)	0.0420 (7)
H6	0.4844	0.1551	0.5286	0.050*
C7	0.1727 (6)	0.0696 (2)	0.4804 (2)	0.0378 (7)
C8	0.0219 (6)	0.0483 (2)	0.3964 (2)	0.0378 (7)
H8	−0.1127	−0.0027	0.4000	0.045*
C9	0.0750 (5)	0.1044 (2)	0.30701 (18)	0.0343 (6)
C10	0.4971 (6)	0.3342 (2)	0.1934 (2)	0.0484 (8)
H10A	0.6610	0.3057	0.2137	0.073*
H10B	0.4508	0.3965	0.2361	0.073*
H10C	0.5053	0.3589	0.1240	0.073*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0424 (12)	0.0494 (11)	0.0358 (10)	−0.0115 (11)	−0.0033 (9)	0.0021 (9)

O2	0.0535 (13)	0.0650 (13)	0.0402 (11)	−0.0092 (12)	−0.0113 (11)	−0.0009 (10)
O3	0.0579 (14)	0.0548 (11)	0.0405 (11)	−0.0058 (12)	−0.0015 (11)	0.0087 (9)
C1	0.0398 (17)	0.0432 (16)	0.0342 (14)	0.0004 (13)	−0.0010 (13)	0.0000 (13)
C2	0.0458 (17)	0.0428 (15)	0.0361 (15)	−0.0024 (14)	0.0051 (14)	0.0037 (12)
C3	0.0359 (16)	0.0343 (13)	0.0391 (15)	0.0002 (13)	0.0080 (14)	−0.0027 (12)
C4	0.0305 (14)	0.0368 (14)	0.0373 (15)	0.0006 (12)	0.0034 (12)	−0.0059 (12)
C5	0.0341 (16)	0.0413 (14)	0.0444 (16)	−0.0075 (13)	0.0003 (15)	−0.0042 (13)
C6	0.0375 (16)	0.0482 (15)	0.0402 (14)	−0.0017 (15)	−0.0049 (14)	−0.0043 (13)
C7	0.0389 (16)	0.0370 (14)	0.0376 (14)	0.0034 (13)	0.0048 (14)	0.0007 (12)
C8	0.0352 (15)	0.0371 (14)	0.0411 (15)	−0.0038 (13)	0.0019 (13)	−0.0007 (12)
C9	0.0324 (15)	0.0366 (14)	0.0340 (13)	−0.0001 (13)	−0.0021 (13)	−0.0044 (12)
C10	0.0506 (17)	0.0442 (16)	0.0505 (17)	−0.0077 (16)	0.0058 (17)	0.0018 (13)

Geometric parameters (Å, °)

O1—C1	1.366 (3)	C4—C5	1.399 (4)
O1—C9	1.378 (3)	C5—C6	1.367 (4)
O2—C1	1.215 (3)	C5—H5	0.9300
O3—C7	1.351 (3)	C6—C7	1.391 (4)
O3—H3	0.8200	C6—H6	0.9300
C1—C2	1.427 (4)	C7—C8	1.381 (4)
C2—C3	1.346 (4)	C8—C9	1.380 (4)
C2—H2	0.9300	C8—H8	0.9300
C3—C4	1.443 (4)	C10—H10A	0.9600
C3—C10	1.498 (4)	C10—H10B	0.9600
C4—C9	1.396 (4)	C10—H10C	0.9600
C1—O1—C9	121.4 (2)	C5—C6—H6	119.9
C7—O3—H3	109.5	C7—C6—H6	119.9
O2—C1—O1	116.5 (2)	O3—C7—C8	122.8 (3)
O2—C1—C2	125.6 (3)	O3—C7—C6	117.2 (2)
O1—C1—C2	117.9 (2)	C8—C7—C6	120.0 (2)
C3—C2—C1	122.8 (2)	C9—C8—C7	118.6 (3)
C3—C2—H2	118.6	C9—C8—H8	120.7
C1—C2—H2	118.6	C7—C8—H8	120.7
C2—C3—C4	118.2 (2)	O1—C9—C8	116.2 (2)
C2—C3—C10	121.6 (2)	O1—C9—C4	120.7 (2)
C4—C3—C10	120.1 (2)	C8—C9—C4	123.0 (2)
C9—C4—C5	116.3 (2)	C3—C10—H10A	109.5
C9—C4—C3	118.8 (2)	C3—C10—H10B	109.5
C5—C4—C3	124.9 (2)	H10A—C10—H10B	109.5
C6—C5—C4	121.8 (3)	C3—C10—H10C	109.5
C6—C5—H5	119.1	H10A—C10—H10C	109.5
C4—C5—H5	119.1	H10B—C10—H10C	109.5
C5—C6—C7	120.2 (3)		
C9—O1—C1—O2	179.5 (2)	C5—C6—C7—O3	−175.9 (3)
C9—O1—C1—C2	−1.7 (4)	C5—C6—C7—C8	3.2 (4)
O2—C1—C2—C3	180.0 (3)	O3—C7—C8—C9	177.1 (3)
O1—C1—C2—C3	1.3 (4)	C6—C7—C8—C9	−1.9 (4)
C1—C2—C3—C4	1.1 (4)	C1—O1—C9—C8	178.3 (3)

C1—C2—C3—C10	−177.5 (3)	C1—O1—C9—C4	−0.4 (4)
C2—C3—C4—C9	−3.1 (4)	C7—C8—C9—O1	−179.9 (2)
C10—C3—C4—C9	175.5 (2)	C7—C8—C9—C4	−1.2 (4)
C2—C3—C4—C5	178.4 (3)	C5—C4—C9—O1	−178.6 (2)
C10—C3—C4—C5	−2.9 (4)	C3—C4—C9—O1	2.9 (4)
C9—C4—C5—C6	−1.4 (4)	C5—C4—C9—C8	2.8 (4)
C3—C4—C5—C6	177.1 (3)	C3—C4—C9—C8	−175.8 (3)
C4—C5—C6—C7	−1.5 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2^{i}	0.82	1.89	2.712 (3)	175
C2—H2···O2^{ii}	0.93	2.51	3.410 (4)	162

Symmetry codes: (i) −*x*−1/2, −*y*, *z*+1/2; (ii) *x*+1/2, −*y*+1/2, −*z*.

Fig. 1

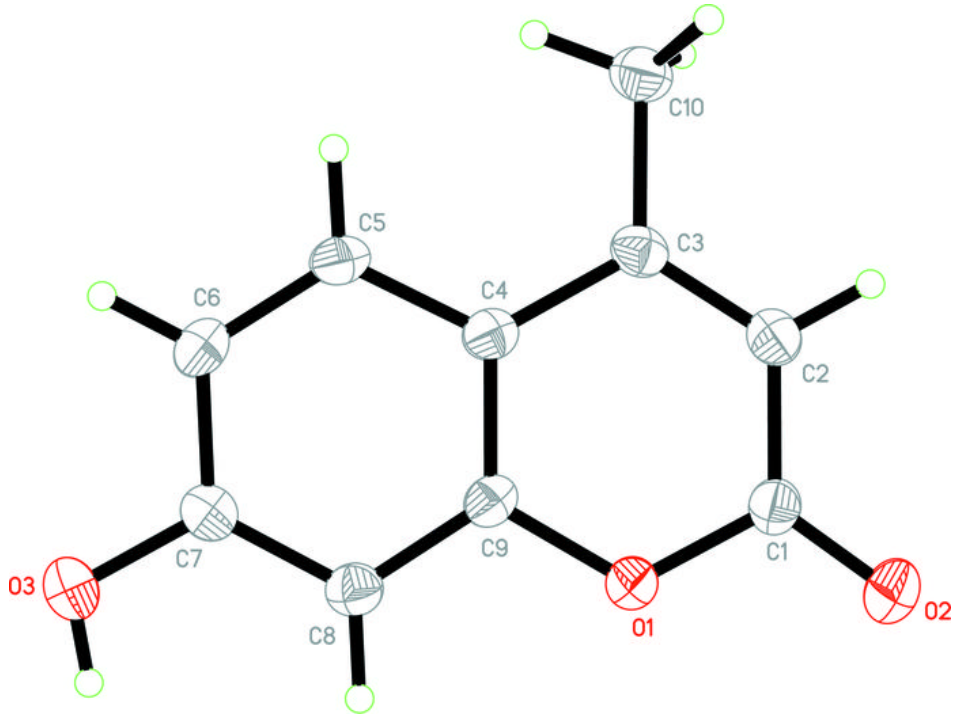


Fig. 2

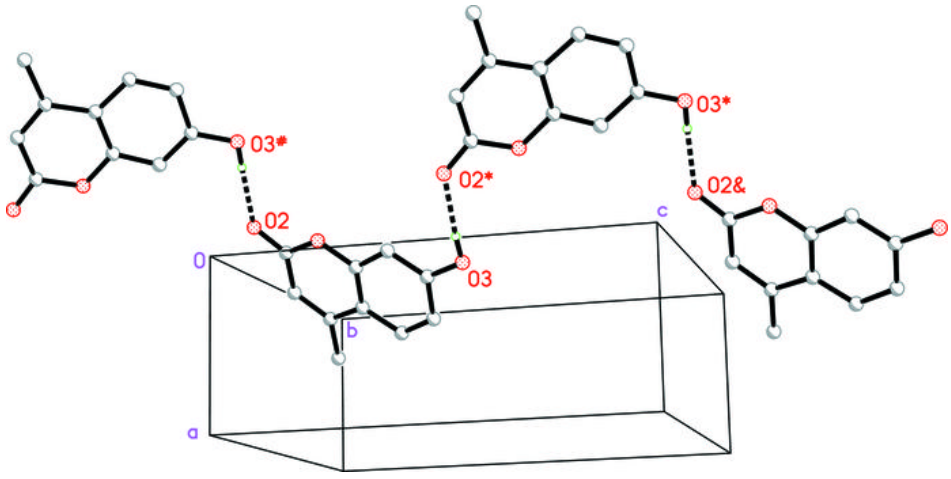


Fig. 3