Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

7-Hydroxy-4-methyl-2*H*-chromen-2-one monohydrate

Ray J. Butcher,[a] Jerry P. Jasinski,[b]* H. S. Yathirajan,[c] B. Narayana[d] and Samshad[d]

[a]Department of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA, [b]Department of Chemistry, Keene State College, 229 Main Street, Keene, NH 03435-2001, USA, [c]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, and [d]Department of Chemistry, Mangalore University, Mangalagangotri 574 199, India
Correspondence e-mail: jjasinski@keene.edu

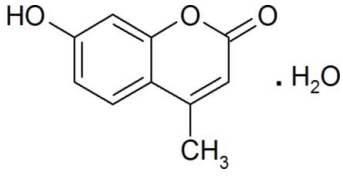
Received 3 July 2007; accepted 4 July 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.052; wR factor = 0.135; data-to-parameter ratio = 20.1.

In the title compound, $C_{10}H_8O_3\cdot H_2O$, the 2*H*-chromen-2-one ring system, also known as coumarin, is planar. Intermolecular O—H···O hydrogen bonding between the solvent water molecule, and the carbonyl and hydroxyl groups of the coumarin ring system links the molecules into a two-dimensional network parallel to the $(10\bar{1})$ plane. In addition, the crystal packing is stabilized by π–π interaction between the coumarin ring systems of the inversion-related molecules stacked along the *a* axis, with a centroid–centroid separation of 3.536 (1) Å.

Related literature

For the synthesis of the title compound, see: Vogel (1989). For related structures, see: Toffoli *et al.* (1985); Bruno *et al.* (2001); Baumer *et al.* (2003, 2004); Zhong *et al.* (2006). For background, see: Pawar & Mulwad (2004); Lin *et al.* (2006); Urano *et al.* (1995); Zhang *et al.* (2003); Aggarwal *et al.* (1996).



Experimental

Crystal data

$C_{10}H_8O_3\cdot H_2O$
M_r = 194.18
Monoclinic, $P2_1/n$
a = 6.9508 (5) Å
b = 11.3074 (8) Å
c = 11.7833 (8) Å
β = 105.643 (1)°
V = 891.81 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 K
0.48 × 0.30 × 0.19 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.794, T_{max} = 1.000
10240 measured reflections
2711 independent reflections
2509 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.135
S = 1.13
2711 reflections
135 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.55 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
O1*W*—H1*W*2···O1^{i}	0.851 (14)	2.014 (15)	2.8509 (13)	168 (2)
O1*W*—H1*W*1···O1	0.819 (14)	1.976 (15)	2.7936 (13)	175 (2)
O2—H2···O1*W*^{ii}	0.84	1.82	2.6583 (12)	174

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 2000).

BN thanks Mangalore University for use of their research facilities. RJB acknowledges the Laboratory for the Structure of Matter at the Naval Research Laboratory for access to their diffractometers.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2411).

References

Aggarwal, M. D., Wang, W. S., Choi, J., Myres, T., Clark, R. D., Penn, B. G., Frazier, D. O. & Sanghadasa, M. (1996). *J. Cryst. Growth*, **166**, 542–544.
Baumer, V. N., Karasev, A. A. & Sizova, Z. A. (2003). *Acta Cryst.* E**59**, o54–o56.
Baumer, V. N., Kovalenko, S. M., Sytnyk, K. M. & Chernykh, V. P. (2004). *Acta Cryst.* E**60**, o1417–o1418.
Bruker (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2006). *APEX2*. Version 2.1-0. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruno, G., Nicoló, F., Rotondo, A., Foti, F., Risitano, F., Grassi, G. & Bilardo, C. (2001). *Acta Cryst.* C**57**, 493–494.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Lin, C. M., Huang, S. T., Lee, F. W., Kuo, H. S. & Lin, M. H. (2006). *Bioorg. Med. Chem.* **14**, 4402–4409.
Pawar, R. B. & Mulwad, V. V. (2004). *Chem. Heterocycl. Compounds*, **440**, 257–264.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2003). *SADABS*. University of Göttingen, Germany.
Toffoli, P., Khodadad, P., Rodier, N., Céolin, R. & Astoin, J. (1985). *Acta Cryst.* C**41**, 933–935.

  doi:10.1107/S1600536807032710

Urano, T., Nagasaka, H., Tsuchiyama, M., Shimizu, S., Kawazoe, K., Shimizu, M. & Yamaoka, T. (1995). *Bull. Chem. Soc. Jpn*, **68**, 1661–1668.
Zhang, Y. Y., Meng, X. M., Wang, X. L. & Xu, L. H. (2003). *Dyes Pigm.* **56**, 189–194.
Zhong, J.-L., Lu, Y. & Zheng, Q.-T. (2006). *Acta Cryst.* E**62**, o697–o698.
Vogel, A. I. (1989). *Vogel's Textbook of Practical Organic Chemistry*, 5th ed., edited by B. S. Furniss, A. J. Hannaford, P. W. G. Smith & A. R. Tatchell, p. 1249. London: Longman.

supplementary materials

Acta Cryst. (2007). E**63**, o3412-o3413 [**doi:10.1107/S1600536807032710**]

7-Hydroxy-4-methyl-2*H*-chromen-2-one monohydrate

R. J. Butcher, J. P. Jasinski, H. S. Yathirajan, B. Narayana and Samshad

Comment

Compounds incorporating benzopyrone structural units posses a wide range of biological activities. A coumarin nucleus is the basis of various compounds possessing anticoagulant and anti-inflammatory activities. 2*H*-2-Chromenone (coumarin) derivatives are widely used for production of highly effective fluorescent dyes for synthetic fibres and daylight fluorescent pigments. These derivatives also play a vital role in electrophotographic and electroluminiscent devices. Synthesis and investigation of new substituted 2*H*-2-chromenone derivatives make possible new ways for scientific and technical usage. The present day demand is for large and high quality ferroelectric, piezoelectric single crystals with minimum defects and inhomogenities. The important goal of crystal growth is the improvement of microscopic and macroscopic homogeneity, which is a necessity for any application. In addition, coumarin derivatives are known as bioactive compounds with weakly toxic, anticarcinogenic, anticoagulant and antibiotic activities. In continuation of our work on crystal structures of organic compounds and in view of the importance of the title compound, we report here its crystal structure.

The non-H atoms of the coumarin derivative are coplanar (Fig. 1), with a maximum deviation of 0.037 (1) Å for atom C4. The hydroxyl group is coplanar with the coumarin ring system. In the crystal structure, the molecules are linked into a two-dimensional network parallel to the (1 0 $\bar{1}$) plane by O—H···O hydrogen bonds involving the water molecules (Fig. 2). In addition π-π stacking interaction is observed between the coumarin ring systems of the inversion related moleules stacked along the *a* axis, with a centroid-centroid separation of 3.536 (1) Å.

Experimental

The title compound was synthesized according to the method reported in the literature (Furniss *et al.*, 1989). The compound was recrystallized from acetone-toluene (2:1 *v*/*v*) (m.p. 457 K).

Refinement

The water H atoms were located in a difference Fourier map and refined with O—H distance restraints of 0.85 (1) Å, and with U_{iso}(H) values of 1.5U_{eq}(O). The remaining H atoms were included in calculated positions (O—H = 0.84 Å and C—H = 0.95 or 0.98 Å) and refined in riding model approximation, with U_{iso}(H) = 1.17–1.48U_{eq}(C).

Figures

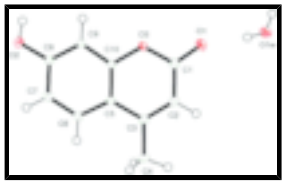


Fig. 1. Molecular structure of the title compound, showing atomic labeling and 50% probability displacement ellipsoids.

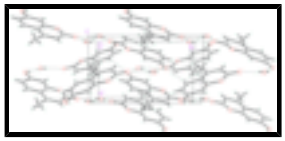


Fig. 2. Packing diagram of the title compound, viewed down the *c* axis. Dashed lines indicate C—H···O hydrogen bonds.

7-Hydroxy-4-methyl-2*H*-chromen-2-one monohydrate

Crystal data

$C_{10}H_8O_3 \cdot H_2O$	$F_{000} = 408$
$M_r = 194.18$	$D_x = 1.446$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 7134 reflections
$a = 6.9508$ (5) Å	$\theta = 2.5$–30.6º
$b = 11.3074$ (8) Å	$\mu = 0.11$ mm^{-1}
$c = 11.7833$ (8) Å	$T = 100$ K
$\beta = 105.643$ (1)º	Chunk, pale yellow
$V = 891.81$ (11) Å^3	0.48 × 0.30 × 0.19 mm
$Z = 4$	

Data collection

Bruker APEXII CCD area-detector diffractometer	2711 independent reflections
Radiation source: fine-focus sealed tube	2509 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.021$
$T = 100$ K	$\theta_{max} = 30.6$º
φ and ω scans	$\theta_{min} = 2.5$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -9 \rightarrow 9$
$T_{min} = 0.794$, $T_{max} = 1.000$	$k = -16 \rightarrow 16$
10240 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.135$	$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.3396P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.13$	$(\Delta/\sigma)_{max} = 0.001$
2711 reflections	$\Delta\rho_{max} = 0.55$ e Å^{-3}
135 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
3 restraints	Extinction correction: none

Primary atom site location: structure-invariant direct methods

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	0.02672 (15)	0.15966 (7)	0.42412 (8)	0.0226 (2)
O2	0.46265 (13)	0.69064 (7)	0.65463 (7)	0.0190 (2)
H2	0.4823	0.6504	0.7166	0.023*
O3	0.16176 (12)	0.33058 (7)	0.49034 (7)	0.01523 (18)
O1W	0.03518 (15)	−0.07764 (8)	0.35966 (8)	0.0228 (2)
H1W1	0.029 (3)	−0.0072 (13)	0.3744 (17)	0.034*
H1W2	0.006 (3)	−0.1104 (15)	0.4179 (15)	0.034*
C1	0.05814 (17)	0.26209 (10)	0.39892 (10)	0.0161 (2)
C2	−0.00631 (17)	0.31431 (10)	0.28374 (10)	0.0166 (2)
H2A	−0.0804	0.2677	0.2198	0.020*
C3	0.03567 (16)	0.42846 (10)	0.26368 (9)	0.0146 (2)
C4	−0.03490 (18)	0.48234 (11)	0.14348 (10)	0.0196 (2)
H4A	−0.1139	0.4242	0.0886	0.029*
H4B	0.0807	0.5063	0.1163	0.029*
H4C	−0.1176	0.5517	0.1469	0.029*
C5	0.14888 (16)	0.49870 (9)	0.36148 (9)	0.0132 (2)
C6	0.20288 (17)	0.61797 (10)	0.35288 (10)	0.0156 (2)
H6A	0.1667	0.6561	0.2783	0.019*
C7	0.30706 (17)	0.68003 (10)	0.45074 (10)	0.0161 (2)
H7A	0.3431	0.7601	0.4430	0.019*
C8	0.36036 (16)	0.62531 (10)	0.56205 (9)	0.0143 (2)
C9	0.30837 (16)	0.50774 (9)	0.57400 (9)	0.0139 (2)
H9A	0.3421	0.4702	0.6489	0.017*
C10	0.20606 (16)	0.44701 (9)	0.47347 (9)	0.0128 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0357 (5)	0.0129 (4)	0.0188 (4)	−0.0039 (3)	0.0067 (4)	−0.0007 (3)
O2	0.0258 (4)	0.0143 (4)	0.0146 (4)	−0.0039 (3)	0.0013 (3)	−0.0018 (3)
O3	0.0224 (4)	0.0105 (3)	0.0124 (4)	−0.0018 (3)	0.0039 (3)	0.0004 (3)

O1W	0.0376 (5)	0.0148 (4)	0.0164 (4)	0.0019 (3)	0.0079 (4)	0.0006 (3)
C1	0.0208 (5)	0.0136 (5)	0.0146 (5)	−0.0004 (4)	0.0059 (4)	−0.0017 (4)
C2	0.0208 (5)	0.0163 (5)	0.0124 (5)	−0.0011 (4)	0.0039 (4)	−0.0021 (4)
C3	0.0158 (5)	0.0169 (5)	0.0111 (4)	0.0011 (4)	0.0039 (3)	−0.0004 (4)
C4	0.0233 (5)	0.0217 (5)	0.0121 (5)	−0.0002 (4)	0.0020 (4)	0.0023 (4)
C5	0.0152 (5)	0.0127 (4)	0.0117 (4)	0.0015 (3)	0.0037 (3)	0.0008 (3)
C6	0.0179 (5)	0.0142 (5)	0.0144 (5)	0.0010 (4)	0.0039 (4)	0.0032 (4)
C7	0.0186 (5)	0.0123 (5)	0.0172 (5)	0.0000 (4)	0.0042 (4)	0.0021 (4)
C8	0.0152 (5)	0.0132 (5)	0.0145 (5)	0.0005 (4)	0.0039 (4)	−0.0009 (4)
C9	0.0173 (5)	0.0132 (4)	0.0115 (4)	0.0007 (4)	0.0041 (4)	0.0007 (3)
C10	0.0158 (5)	0.0100 (4)	0.0129 (4)	0.0009 (3)	0.0043 (4)	0.0010 (3)

Geometric parameters (Å, °)

O1—C1	1.2298 (14)	C4—H4A	0.98
O2—C8	1.3501 (13)	C4—H4B	0.98
O2—H2	0.84	C4—H4C	0.98
O3—C1	1.3641 (13)	C5—C10	1.3993 (14)
O3—C10	1.3786 (12)	C5—C6	1.4108 (15)
O1W—H1W1	0.819 (14)	C6—C7	1.3770 (15)
O1W—H1W2	0.851 (14)	C6—H6A	0.95
C1—C2	1.4365 (15)	C7—C8	1.4065 (15)
C2—C3	1.3579 (15)	C7—H7A	0.95
C2—H2A	0.95	C8—C9	1.3947 (15)
C3—C5	1.4454 (15)	C9—C10	1.3879 (14)
C3—C4	1.4978 (15)	C9—H9A	0.95
C8—O2—H2	109.5	C10—C5—C6	116.98 (10)
C1—O3—C10	121.28 (8)	C10—C5—C3	118.52 (9)
H1W1—O1W—H1W2	102.4 (16)	C6—C5—C3	124.49 (10)
O1—C1—O3	115.74 (10)	C7—C6—C5	121.13 (10)
O1—C1—C2	125.80 (10)	C7—C6—H6A	119.4
O3—C1—C2	118.46 (10)	C5—C6—H6A	119.4
C3—C2—C1	121.89 (10)	C6—C7—C8	120.24 (10)
C3—C2—H2A	119.1	C6—C7—H7A	119.9
C1—C2—H2A	119.1	C8—C7—H7A	119.9
C2—C3—C5	118.68 (10)	O2—C8—C9	122.30 (10)
C2—C3—C4	121.35 (10)	O2—C8—C7	117.49 (10)
C5—C3—C4	119.97 (10)	C9—C8—C7	120.21 (10)
C3—C4—H4A	109.5	C10—C9—C8	118.20 (10)
C3—C4—H4B	109.5	C10—C9—H9A	120.9
H4A—C4—H4B	109.5	C8—C9—H9A	120.9
C3—C4—H4C	109.5	O3—C10—C9	115.62 (9)
H4A—C4—H4C	109.5	O3—C10—C5	121.15 (9)
H4B—C4—H4C	109.5	C9—C10—C5	123.23 (10)
C10—O3—C1—O1	−179.87 (10)	C6—C7—C8—O2	179.91 (10)
C10—O3—C1—C2	−0.59 (15)	C6—C7—C8—C9	0.13 (17)
O1—C1—C2—C3	−179.79 (12)	O2—C8—C9—C10	−178.93 (10)
O3—C1—C2—C3	1.00 (17)	C7—C8—C9—C10	0.84 (16)
C1—C2—C3—C5	0.13 (17)	C1—O3—C10—C9	178.66 (10)

C1—C2—C3—C4	−179.12 (11)	C1—O3—C10—C5	−0.98 (15)
C2—C3—C5—C10	−1.65 (16)	C8—C9—C10—O3	178.91 (9)
C4—C3—C5—C10	177.62 (10)	C8—C9—C10—C5	−1.46 (17)
C2—C3—C5—C6	179.90 (10)	C6—C5—C10—O3	−179.33 (9)
C4—C3—C5—C6	−0.84 (17)	C3—C5—C10—O3	2.09 (15)
C10—C5—C6—C7	−0.03 (16)	C6—C5—C10—C9	1.06 (16)
C3—C5—C6—C7	178.45 (10)	C3—C5—C10—C9	−177.51 (10)
C5—C6—C7—C8	−0.54 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W2···O1^{i}	0.851 (14)	2.014 (15)	2.8509 (13)	168 (2)
O1W—H1W1···O1	0.819 (14)	1.976 (15)	2.7936 (13)	175 (2)
O2—H2···O1W^{ii}	0.84	1.82	2.6583 (12)	174

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

Fig. 1

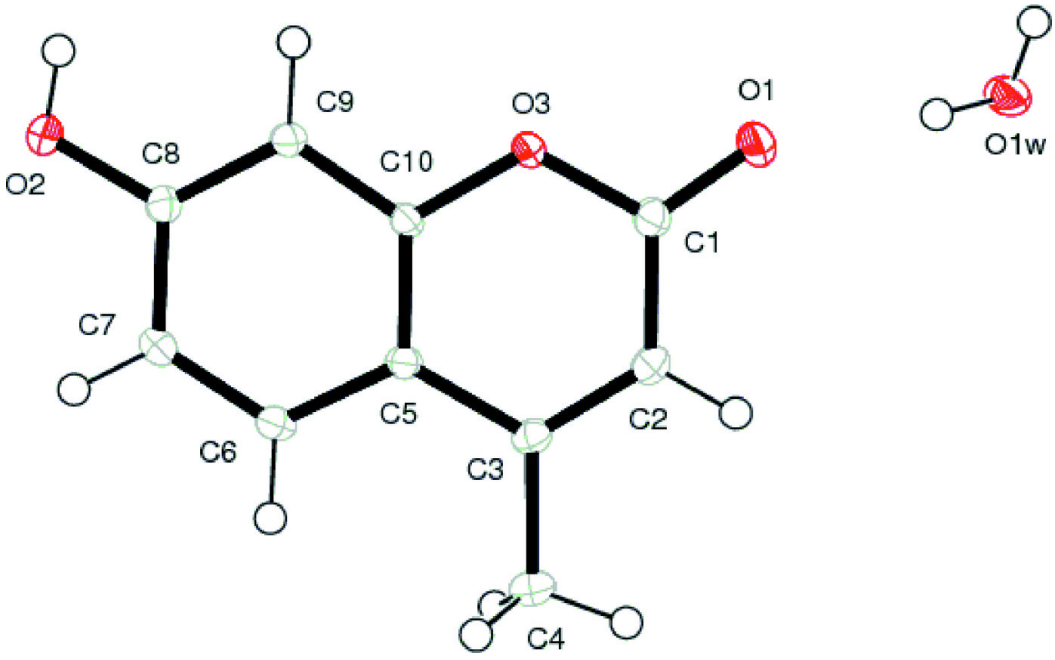


Fig. 2