

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

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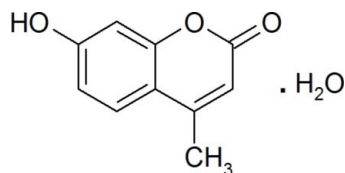
Received 3 July 2007; accepted 4 July 2007

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.052; wR factor = 0.135; data-to-parameter ratio = 20.1.

In the title compound, $\text{C}_{10}\text{H}_8\text{O}_3 \cdot \text{H}_2\text{O}$, the 2H-chromen-2-one ring system, also known as coumarin, is planar. Intermolecular $\text{O}-\text{H} \cdots \text{O}$ 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 $\pi-\pi$ 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

$\text{C}_{10}\text{H}_8\text{O}_3 \cdot \text{H}_2\text{O}$
 $M_r = 194.18$
Monoclinic, $P2_1/n$
 $a = 6.9508$ (5) Å
 $b = 11.3074$ (8) Å
 $c = 11.7833$ (8) Å

$\beta = 105.643$ (1)°
 $V = 891.81$ (11) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 100$ K

$0.48 \times 0.30 \times 0.19$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

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, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O1W}-\text{H1W2} \cdots \text{O1}^i$	0.851 (14)	2.014 (15)	2.8509 (13)	168 (2)
$\text{O1W}-\text{H1W1} \cdots \text{O1}$	0.819 (14)	1.976 (15)	2.7936 (13)	175 (2)
$\text{O2}-\text{H2} \cdots \text{O1W}^{\text{ii}}$	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).

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supplementary materials

Acta Cryst. (2007). E63, o3412-o3413 [doi:10.1107/S1600536807032710]

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

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Comment

Compounds incorporating benzopyrone structural units possess a wide range of biological activities. A coumarin nucleus is the basis of various compounds possessing anticoagulant and anti-inflammatory activities. 2H-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 electroluminescent devices. Synthesis and investigation of new substituted 2H-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 inhomogeneities. 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 molecules 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 $U_{\text{iso}}(\text{H})$ values of $1.5U_{\text{eq}}(\text{O})$. 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 $U_{\text{iso}}(\text{H}) = 1.17\text{--}1.48U_{\text{eq}}(\text{C})$.

Figures

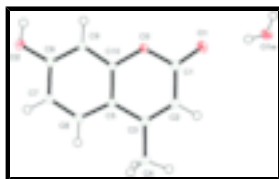


Fig. 1. Molecular structure of the title compound, showing atomic labeling and 50% probability displacement ellipsoids.

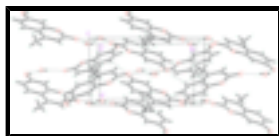


Fig. 2. Packing diagram of the title compound, viewed down the *c* axis. Dashed lines indicate C—H...O hydrogen bonds.

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

Crystal data

$C_{10}H_8O_3 \cdot H_2O$	$F_{000} = 408$
$M_r = 194.18$	$D_x = 1.446 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
Hall symbol: $-P\ 2_1/n$	$\lambda = 0.71073 \text{ \AA}$
$a = 6.9508 (5) \text{ \AA}$	Cell parameters from 7134 reflections
$b = 11.3074 (8) \text{ \AA}$	$\theta = 2.5\text{--}30.6^\circ$
$c = 11.7833 (8) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$\beta = 105.643 (1)^\circ$	$T = 100 \text{ K}$
$V = 891.81 (11) \text{ \AA}^3$	Chunk, pale yellow
$Z = 4$	$0.48 \times 0.30 \times 0.19 \text{ mm}$

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	$R_{\text{int}} = 0.021$
$T = 100 \text{ K}$	$\theta_{\text{max}} = 30.6^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.5^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.794$, $T_{\text{max}} = 1.000$	$k = -16 \rightarrow 16$
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
$wR(F^2) = 0.135$	$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.3396P]$
$S = 1.13$	where $P = (F_o^2 + 2F_c^2)/3$
2711 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
135 parameters	$\Delta\rho_{\text{max}} = 0.55 \text{ e \AA}^{-3}$
3 restraints	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
	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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^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)

supplementary materials

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, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1W—H1W2 \cdots O1 ⁱ	0.851 (14)	2.014 (15)	2.8509 (13)	168 (2)
O1W—H1W1 \cdots O1	0.819 (14)	1.976 (15)	2.7936 (13)	175 (2)
O2—H2 \cdots 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

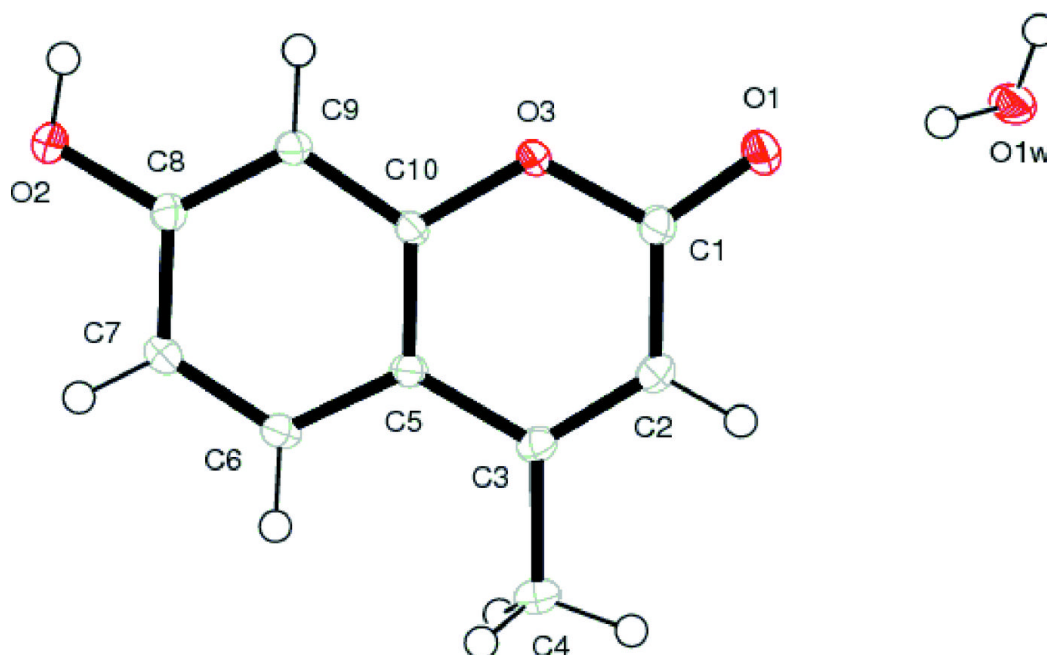


Fig. 2

