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8-Acetyl-4-methyl-2-oxo-2H-chromen-7-yl acetate

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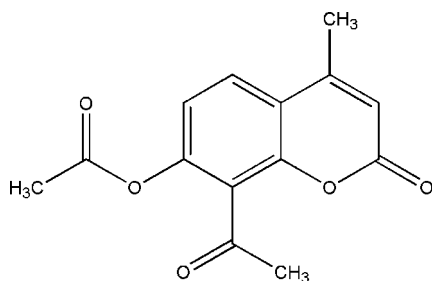
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.048; wR factor = 0.150; data-to-parameter ratio = 13.1.

In the title compound, $\text{C}_{14}\text{H}_{12}\text{O}_5$, the benzopyran-2-one ring system is approximately planar [maximum deviation = $0.018(1)$ Å]; the mean plane is oriented at dihedral angles of $52.26(11)$ and $72.92(7)^\circ$, respectively, to the acetyl and acetoxy groups. In the crystal, π - π stacking is observed between parallel benzene rings of adjacent molecules, the centroid-centroid distance being $3.6774(17)$ Å. Intermolecular weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding, and $\text{C}=\text{O}\cdots\text{C}=\text{O}$ [$\text{O}\cdots\text{C} = 3.058(3)$ Å] and $\text{C}=\text{O}\cdots\pi$ [$\text{O}\cdots\text{centroid} = 3.328(2)$ Å] interactions occur in the crystal structure.

Related literature

For structures of related coumarin derivatives, see: Yang *et al.* (2006, 2007, 2010).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_{12}\text{O}_5$ $M_r = 260.24$

Triclinic, $P\bar{1}$
 $a = 8.198(3)$ Å
 $b = 8.504(3)$ Å
 $c = 9.644(3)$ Å
 $\alpha = 90.213(4)^\circ$
 $\beta = 97.761(4)^\circ$
 $\gamma = 111.686(4)^\circ$

$V = 618.0(3)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 298$ K
 $0.30 \times 0.10 \times 0.10$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.969$, $T_{\max} = 0.989$

4891 measured reflections
 2292 independent reflections
 1572 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.150$
 $S = 1.07$
 2292 reflections

175 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C6}-\text{H6}\cdots\text{O2}^i$	0.93	2.52	3.324 (3)	145

Symmetry code: (i) $x - 1, y - 1, z$.

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg & Berndt, 1999); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5385).

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supporting information

Acta Cryst. (2011). E67, o3382 [https://doi.org/10.1107/S1600536811048835]

8-Acetyl-4-methyl-2-oxo-2H-chromen-7-yl acetate**Shu-Ping Yang, Li-Jun Han, Xin-Ran He and Li-Juan Chen****S1. Comment**

Previous we have reported the crystal structures of some coumarin derivatives (Yang *et al.*, 2006, 2007, 2010). As part of our study of the crystal structures of coumarin derivatives, we described here the crystal structure of 8-acetyl-7-acetoxy-4-methyl-2H-1-benzopyran-2-one, (I).

In the molecule (I) (Fig. 1), the benzopyran-2-one ring system is approximately planar [maximum deviation 0.018 (1) Å]; the mean plane is oriented with respect to the acetyl and acetoxy groups at 52.26 (11) and 72.92 (7)°, respectively.

Molecules are linked together by one weak C—H···O hydrogen bond (Table. 1), two C=O···C=O interactions: C1=O2···C13ⁱⁱ=O4ⁱⁱ [O2···C13ⁱⁱ = 3.101 (3) Å, C1=O2···C13ⁱⁱ = 143.37 (15)°; symmetry code:(ii). $x + 1, y + 1, z$] and C1=O2···C1ⁱⁱⁱ=O2ⁱⁱⁱ [O2···C1ⁱⁱⁱ = 3.058 (3) Å, C1=O2···C1ⁱⁱⁱ = 88.63 (13)°; symmetry code: (iii). $1 - x, 2 - y, 2 - z$.], one C=O··· π interaction C1=O2···Cg1ⁱⁱⁱ [O2···Cg1ⁱⁱⁱ = 3.328 (2) Å, C1=O2···Cg1ⁱⁱⁱ = 113.74 (14)°, Cg1 is the centroid of the pyran ring] and one π - π stacking interaction Cg2···Cg2^{iv} [Cg2···Cg2^{iv} = 3.6774 (17) Å, Cg2 is the centroid of the benzene ring; symmetry code: (iv) $-x, 1 - y, 2 - z$] and generated a two-dimensional crystal structure by translation and inversion symmetry.

S2. Experimental

To a solution containing 8-acetyl-7-hydroxy-4-methylcoumarin (2.18 g, 10 mmol) and anhydrous pyridine (10 ml), a solution of acetic anhydride (1.53 g, 15 mmol) was slowly added at 278–283 K, with stirring, then the reaction mixture was stirred at 303 K continuously for 24 h and then poured into ice–water (200 ml). The solid obtained was filtered off, washed with water and dried at room temperature. Colourless crystal suitable for X-ray structure analysis were obtained by recrystallizing the crude product from an ethanol solution [m.p. 484–485K].

S3. Refinement

All H-atoms were placed in calculated positions (C—H = 0.93 and 0.96 Å) and were included in the refinement in the riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{aromatic C})$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl C})$.

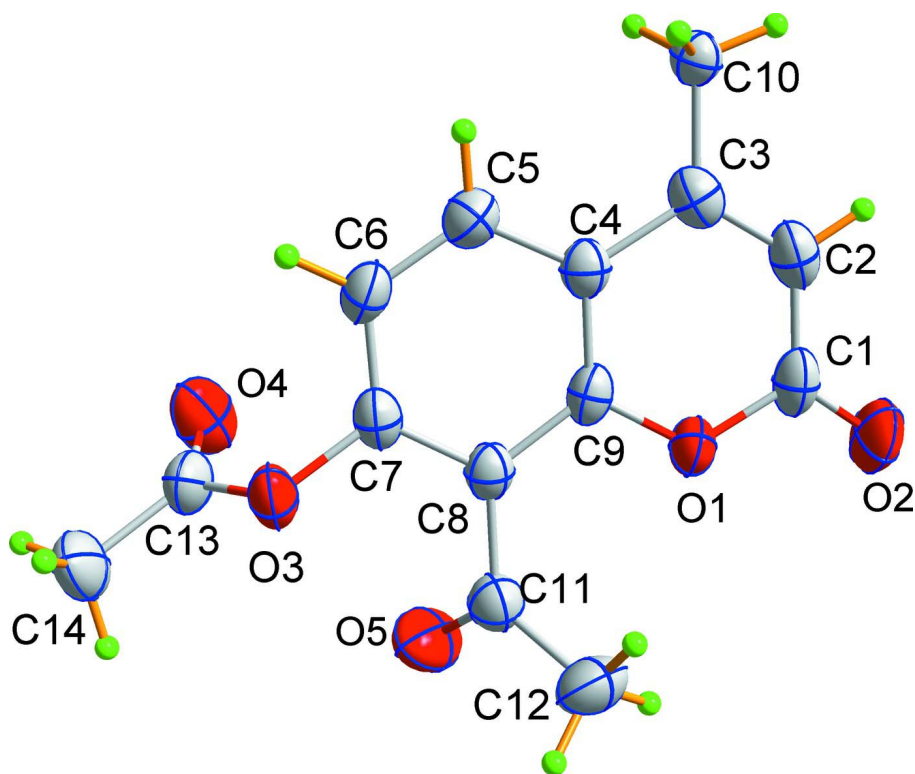


Figure 1

The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

8-Acetyl-4-methyl-2-oxo-2H-chromen-7-yl acetate

Crystal data

$C_{14}H_{12}O_5$

$M_r = 260.24$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 8.198\ (3)\ \text{\AA}$

$b = 8.504\ (3)\ \text{\AA}$

$c = 9.644\ (3)\ \text{\AA}$

$\alpha = 90.213\ (4)^\circ$

$\beta = 97.761\ (4)^\circ$

$\gamma = 111.686\ (4)^\circ$

$V = 618.0\ (3)\ \text{\AA}^3$

$Z = 2$

$F(000) = 272$

$D_x = 1.399\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1283 reflections

$\theta = 2.6\text{--}25.2^\circ$

$\mu = 0.11\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Prism, colourless

$0.30 \times 0.10 \times 0.10\ \text{mm}$

Data collection

Bruker APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2001)

$T_{\min} = 0.969$, $T_{\max} = 0.989$

4891 measured reflections

2292 independent reflections

1572 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 25.5^\circ$, $\theta_{\min} = 2.6^\circ$

$h = -9 \rightarrow 9$

$k = -10 \rightarrow 10$

$l = -11 \rightarrow 11$

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.048$ $wR(F^2) = 0.150$ $S = 1.07$

2292 reflections

175 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0824P)^2 + 0.0107P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$ *Special details*

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3325 (3)	1.0614 (2)	0.9868 (2)	0.0430 (5)
C2	0.2915 (3)	1.0571 (3)	1.1282 (2)	0.0463 (5)
H2	0.3657	1.1427	1.1938	0.056*
C3	0.1515 (3)	0.9353 (2)	1.1690 (2)	0.0409 (5)
C4	0.0343 (2)	0.8014 (2)	1.0672 (2)	0.0354 (5)
C5	-0.1181 (3)	0.6703 (2)	1.0954 (2)	0.0408 (5)
H5	-0.1486	0.6655	1.1852	0.049*
C6	-0.2241 (3)	0.5482 (2)	0.9946 (2)	0.0419 (5)
H6	-0.3251	0.4615	1.0156	0.050*
C7	-0.1786 (2)	0.5559 (2)	0.8607 (2)	0.0368 (5)
C8	-0.0288 (2)	0.6828 (2)	0.8251 (2)	0.0349 (5)
C9	0.0747 (2)	0.8046 (2)	0.9306 (2)	0.0344 (5)
C10	0.1127 (3)	0.9338 (3)	1.3169 (2)	0.0646 (7)
H10A	0.2010	1.0299	1.3709	0.097*
H10B	0.1145	0.8314	1.3574	0.097*
H10C	-0.0023	0.9390	1.3168	0.097*
C11	0.0155 (3)	0.6857 (2)	0.6786 (2)	0.0432 (5)
C12	0.1973 (3)	0.6967 (3)	0.6594 (3)	0.0593 (7)
H12A	0.1881	0.6088	0.5925	0.089*
H12B	0.2594	0.6830	0.7475	0.089*
H12C	0.2612	0.8052	0.6259	0.089*
C13	-0.4432 (3)	0.4153 (3)	0.7065 (2)	0.0463 (5)
C14	-0.5350 (3)	0.2647 (3)	0.6076 (3)	0.0634 (7)
H14A	-0.6577	0.2494	0.5840	0.095*
H14B	-0.5263	0.1661	0.6508	0.095*

H14C	-0.4803	0.2813	0.5241	0.095*
O1	0.21819 (16)	0.93313 (16)	0.89181 (14)	0.0416 (4)
O2	0.4560 (2)	1.16541 (18)	0.94235 (18)	0.0601 (5)
O3	-0.27782 (17)	0.42394 (16)	0.76069 (15)	0.0447 (4)
O4	-0.5014 (2)	0.5178 (2)	0.73698 (19)	0.0684 (5)
O5	-0.0952 (2)	0.6742 (2)	0.57939 (17)	0.0694 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0312 (11)	0.0310 (11)	0.0618 (14)	0.0074 (9)	0.0020 (10)	0.0004 (10)
C2	0.0374 (12)	0.0396 (11)	0.0551 (14)	0.0110 (9)	-0.0060 (10)	-0.0082 (10)
C3	0.0402 (11)	0.0389 (11)	0.0420 (12)	0.0154 (9)	-0.0016 (9)	-0.0018 (9)
C4	0.0314 (10)	0.0343 (10)	0.0405 (11)	0.0132 (8)	0.0027 (8)	0.0019 (8)
C5	0.0393 (11)	0.0409 (11)	0.0417 (11)	0.0134 (9)	0.0093 (9)	0.0056 (9)
C6	0.0312 (10)	0.0360 (11)	0.0529 (13)	0.0047 (8)	0.0103 (9)	0.0043 (9)
C7	0.0292 (10)	0.0314 (10)	0.0463 (12)	0.0091 (8)	0.0000 (8)	-0.0022 (8)
C8	0.0296 (10)	0.0331 (10)	0.0421 (11)	0.0125 (8)	0.0034 (8)	0.0007 (8)
C9	0.0248 (9)	0.0308 (10)	0.0463 (12)	0.0090 (8)	0.0046 (8)	0.0050 (8)
C10	0.0727 (17)	0.0653 (16)	0.0455 (14)	0.0164 (13)	0.0016 (12)	-0.0083 (12)
C11	0.0415 (12)	0.0403 (11)	0.0456 (12)	0.0133 (9)	0.0050 (10)	-0.0001 (9)
C12	0.0539 (14)	0.0754 (16)	0.0571 (15)	0.0293 (12)	0.0221 (12)	0.0068 (12)
C13	0.0319 (11)	0.0500 (12)	0.0508 (13)	0.0092 (10)	0.0026 (9)	-0.0017 (10)
C14	0.0442 (14)	0.0689 (16)	0.0619 (16)	0.0076 (12)	-0.0036 (12)	-0.0196 (13)
O1	0.0315 (7)	0.0372 (8)	0.0489 (9)	0.0040 (6)	0.0070 (6)	0.0021 (6)
O2	0.0442 (9)	0.0414 (9)	0.0804 (12)	-0.0021 (7)	0.0141 (8)	0.0035 (8)
O3	0.0315 (8)	0.0380 (8)	0.0578 (9)	0.0075 (6)	0.0004 (6)	-0.0097 (7)
O4	0.0503 (10)	0.0742 (12)	0.0835 (13)	0.0333 (9)	-0.0093 (9)	-0.0180 (10)
O5	0.0583 (11)	0.1014 (14)	0.0466 (10)	0.0311 (10)	-0.0026 (8)	-0.0036 (9)

Geometric parameters (Å, °)

C1—O2	1.204 (2)	C9—O1	1.374 (2)
C1—O1	1.383 (2)	C10—H10A	0.9600
C1—C2	1.445 (3)	C10—H10B	0.9600
C2—C3	1.338 (3)	C10—H10C	0.9600
C2—H2	0.9300	C11—O5	1.203 (2)
C3—C4	1.453 (3)	C11—C12	1.495 (3)
C3—C10	1.502 (3)	C12—H12A	0.9600
C4—C5	1.394 (3)	C12—H12B	0.9600
C4—C9	1.400 (3)	C12—H12C	0.9600
C5—C6	1.369 (3)	C13—O4	1.191 (2)
C5—H5	0.9300	C13—O3	1.361 (2)
C6—C7	1.388 (3)	C13—C14	1.483 (3)
C6—H6	0.9300	C14—H14A	0.9600
C7—C8	1.388 (3)	C14—H14B	0.9600
C7—O3	1.399 (2)	C14—H14C	0.9600
C8—C9	1.392 (3)	O2—C1 ⁱ	3.058 (3)

C8—C11	1.504 (3)	O2—C13 ⁱⁱ	3.101 (3)
O2—C1—O1	116.15 (19)	C3—C10—H10B	109.5
O2—C1—C2	126.87 (19)	H10A—C10—H10B	109.5
O1—C1—C2	116.97 (17)	C3—C10—H10C	109.5
C3—C2—C1	122.88 (19)	H10A—C10—H10C	109.5
C3—C2—H2	118.6	H10B—C10—H10C	109.5
C1—C2—H2	118.6	O5—C11—C12	121.1 (2)
C2—C3—C4	118.98 (19)	O5—C11—C8	120.33 (19)
C2—C3—C10	121.69 (19)	C12—C11—C8	118.50 (18)
C4—C3—C10	119.33 (19)	C11—C12—H12A	109.5
C5—C4—C9	117.28 (18)	C11—C12—H12B	109.5
C5—C4—C3	124.53 (18)	H12A—C12—H12B	109.5
C9—C4—C3	118.19 (18)	C11—C12—H12C	109.5
C6—C5—C4	121.83 (19)	H12A—C12—H12C	109.5
C6—C5—H5	119.1	H12B—C12—H12C	109.5
C4—C5—H5	119.1	O4—C13—O3	122.68 (19)
C5—C6—C7	118.96 (18)	O4—C13—C14	126.4 (2)
C5—C6—H6	120.5	O3—C13—C14	110.89 (18)
C7—C6—H6	120.5	C13—C14—H14A	109.5
C6—C7—C8	122.35 (18)	C13—C14—H14B	109.5
C6—C7—O3	119.13 (17)	H14A—C14—H14B	109.5
C8—C7—O3	118.30 (17)	C13—C14—H14C	109.5
C7—C8—C9	116.78 (18)	H14A—C14—H14C	109.5
C7—C8—C11	120.47 (17)	H14B—C14—H14C	109.5
C9—C8—C11	122.75 (17)	C9—O1—C1	121.70 (15)
O1—C9—C8	115.92 (17)	C1—O2—C1 ⁱ	88.63 (13)
O1—C9—C4	121.25 (17)	C1—O2—C13 ⁱⁱ	143.37 (15)
C8—C9—C4	122.81 (18)	C1 ⁱ —O2—C13 ⁱⁱ	118.73 (8)
C3—C10—H10A	109.5	C13—O3—C7	117.16 (14)
O2—C1—C2—C3	-179.5 (2)	C3—C4—C9—O1	-1.9 (3)
O1—C1—C2—C3	0.7 (3)	C5—C4—C9—C8	-0.8 (3)
C1—C2—C3—C4	-0.3 (3)	C3—C4—C9—C8	-179.87 (16)
C1—C2—C3—C10	179.75 (19)	C7—C8—C11—O5	50.4 (3)
C2—C3—C4—C5	-178.21 (18)	C9—C8—C11—O5	-129.3 (2)
C10—C3—C4—C5	1.7 (3)	C7—C8—C11—C12	-127.4 (2)
C2—C3—C4—C9	0.8 (3)	C9—C8—C11—C12	52.9 (3)
C10—C3—C4—C9	-179.22 (18)	C8—C9—O1—C1	-179.47 (15)
C9—C4—C5—C6	0.5 (3)	C4—C9—O1—C1	2.4 (3)
C3—C4—C5—C6	179.52 (17)	O2—C1—O1—C9	178.42 (16)
C4—C5—C6—C7	-0.2 (3)	C2—C1—O1—C9	-1.7 (3)
C5—C6—C7—C8	0.2 (3)	O1—C1—O2—C1 ⁱ	-87.95 (16)
C5—C6—C7—O3	174.65 (16)	C2—C1—O2—C1 ⁱ	92.2 (2)
C6—C7—C8—C9	-0.4 (3)	O1—C1—O2—C13 ⁱⁱ	52.7 (3)
O3—C7—C8—C9	-174.95 (15)	C2—C1—O2—C13 ⁱⁱ	-127.1 (2)
C6—C7—C8—C11	179.84 (18)	O4—C13—O3—C7	2.9 (3)
O3—C7—C8—C11	5.3 (3)	C14—C13—O3—C7	-177.22 (18)

C7—C8—C9—O1	-177.37 (14)	C6—C7—O3—C13	73.3 (2)
C11—C8—C9—O1	2.3 (3)	C8—C7—O3—C13	-112.06 (19)
C7—C8—C9—C4	0.7 (3)	C1—O2—C13 ⁱⁱ —O4 ⁱⁱ	69.0 (3)
C11—C8—C9—C4	-179.55 (17)	C1—O2—C1 ⁱ —O2 ⁱ	0.0
C5—C4—C9—O1	177.26 (15)		

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

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
C6—H6...O2 ⁱⁱⁱ	0.93	2.52	3.324 (3)	145

Symmetry code: (iii) $x-1, y-1, z$.