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3-Acetyl-2-hydroxy-2-methylchromene

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In the title compound, $C_{12}H_{12}O_3$, the pyran ring adopts a half-chair conformation. In the crystal, the inversion-related molecules exist as O—H···O hydrogen-bonded dimers and these dimeric pairs are reinforced by C—H···O interactions.

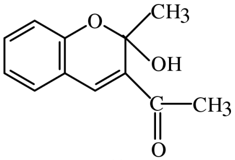
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.067
wR factor = 0.198
Data-to-parameter ratio = 14.9

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

Comment

Chromene derivatives exhibit antifeedant activity against larvae of *Spilarctia obliqua* (Agarwal *et al.*, 2000) and some 2*H*-chromene derivatives are found to possess antimicrobial activities (El-Gaby *et al.*, 2000). Endothelin-A (ET_A) selective receptor antagonists contain the 2*H*-chromene moiety (Ishizuka *et al.*, 2002). In addition to these activities, chromene derivatives possess photochromic properties. The structure determination of the title compound, (I), was undertaken as part of our studies on chromene derivatives.



(I)

In (I) (Fig. 1), the pyran ring adopts a half-chair conformation, with asymmetry parameter Δ_2(C2—O1) = 0.009 (1) (Nardelli, 1983). Atoms O1 and C2 deviate from the weighted least-squares plane through the remaining four atoms of the pyran ring by −0.253 (1) and 0.214 (2) Å, respectively. The hydroxy and methyl groups at C2 adopt pseudo-axial and pseudo-equatorial orientations, respectively. The acetyl group is planar and makes a dihedral angle of 28.6 (1)° with the mean plane passing through atoms C3—C10. The bond angles at sp^3-hybridized atom C2 show substantial distortion from the ideal tetrahedral value of 109.5°. Similar observations have been noted in the crystal structures of related chromene derivatives (Aldoshin *et al.*, 1995, 1996; Yogavel *et al.*, 2003). The C3—C4 [1.343 (2) Å] bond length is comparable with the previously reported value of 1.350 (4) Å for 3-benzoyl-2-hydroxy-2-methylchromene (Yogavel *et al.*, 2003).

The distinctive feature of the crystal structure of (I) is that the inversion-related intermolecular O—H···O hydrogen bonds (Table 2) lead to the formation of dimeric pairs with graph-set $R_2^2(12)$ (Etter *et al.*, 1990), and these dimeric pairs are further reinforced by weak C—H···O interactions.

Experimental

Acetylacetone (10 mmol) in ethanol was mixed with salicylaldehyde (10 mmol), to which was added 0.5 ml of piperidine. The solution

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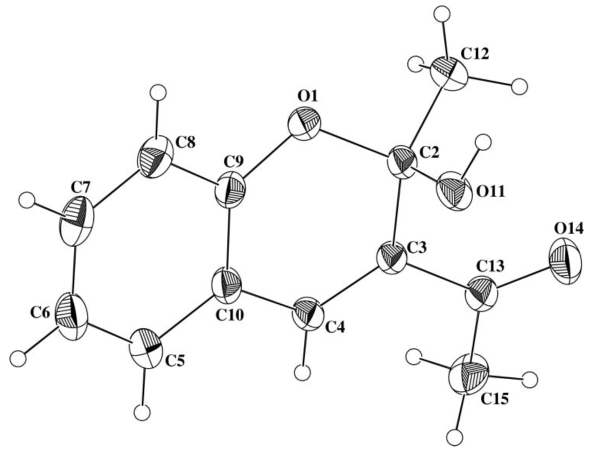

Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 35% probability displacement ellipsoids.

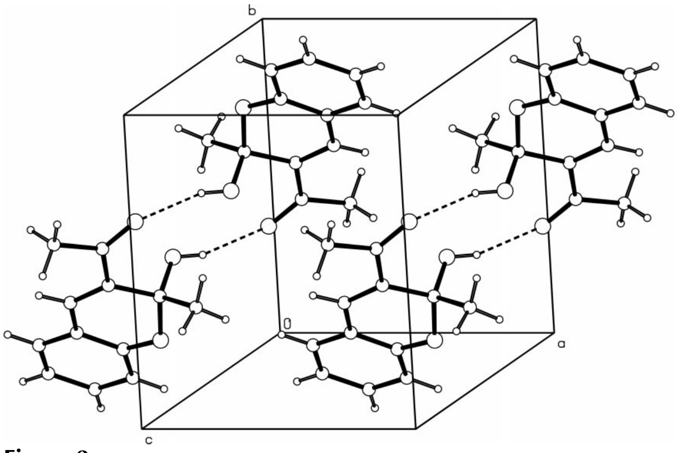

Figure 2
A view of the hydrogen-bonded dimers.

mixture was stirred thoroughly for about 3 h with occasional cooling. This mixture was then kept in a refrigerator for 12 h. A yellow product was obtained and this was separated out, filtered, washed with a small amount of ethanol and dried under vacuum. The title compound was recrystallized from a chloroform/petroleum ether (1:1 volume ratio) solution.

Crystal data

$C_{12}H_{12}O_3$
$M_r = 204.22$
Triclinic, $P\overline{1}$
$a = 7.574$ (2) Å
$b = 8.672$ (2) Å
$c = 8.789$ (3) Å
$\alpha = 106.32$ (3)°
$\beta = 110.99$ (3)°
$\gamma = 93.28$ (3)°
$V = 509.2$ (2) Å^3
$Z = 2$
$D_x = 1.332$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10$–35°
$\mu = 0.79$ mm^{-1}
$T = 293$ (2) K
Prism, yellow
0.25 × 0.25 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: none
2091 measured reflections
2091 independent reflections
1963 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 74.9°$
$h = -8 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -10 \rightarrow 0$
2 standard reflections every 60 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.198$
$S = 1.05$
2091 reflections
140 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1509P)^2 + 0.0906P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.32$ e Å^{-3}
$\Delta\rho_{\min} = -0.33$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.104 (12)

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.364 (2)	C3—C4	1.343 (2)
O1—C2	1.4454 (18)	C13—O14	1.218 (2)
C2—O11	1.3998 (19)		
O11—C2—O1	109.02 (13)	O11—C2—C3	105.64 (12)
O11—C2—C12	114.01 (13)	O1—C2—C3	110.84 (11)
O1—C2—C12	102.54 (13)	C12—C2—C3	114.79 (13)
C9—O1—C2—C3	39.03 (18)	C3—C4—C10—C9	10.4 (2)
O1—C2—C3—C4	−24.25 (19)	C4—C3—C13—O14	−155.77 (17)
C2—C3—C4—C10	0.9 (2)	C2—C3—C13—O14	14.3 (2)
C2—O1—C9—C10	−30.7 (2)	C4—C3—C13—C15	22.5 (2)
O1—C9—C10—C4	4.4 (2)	C2—C3—C13—C15	−167.43 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
O11—H11···O14$^{\rm i}$	0.82	2.15	2.899 (2)	152
C12—H12C···O11$^{\rm i}$	0.96	2.60	3.357 (2)	136
C12—H12C···O14	0.96	2.44	2.990 (2)	116

Symmetry code: (i) $2 - x, 1 - y, 1 - z$.

The H atoms were positioned geometrically and were treated as riding on their parent C and O atoms, with aromatic C—H distances of 0.93 Å, methyl C—H distances of 0.96 Å and O—H distance of 0.82 Å. Rotating-group refinement was used for the methyl and hydroxy groups.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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