Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Benzoyl-2-hydroxy-2-methylchromene

M. Yogavel,[a] D. Velmurugan,[a] K. Sekar,[b] H. Schenk,[c] J. Fraanje,[c] R. Peschar,[c] S. Srinivasan,[d] P. R. Athappan[d] and Z. A. Rafi[e]*

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [b]Bioinformatics Centre and Supercomputer Education and Research Centre, Indian Institute of Science, Bangalore 560 012, India, [c]Laboratory of Crystallography, Institute of Molecular Chemistry, University of Amsterdam, Achtergracht 116, 1018 WV, Amsterdam, The Netherlands, [d]School of Chemistry, Madurai Kamaraj University, Madurai 625 021, India, and [e]Bioinformatics Centre, School of Biotechnology, Madurai Kamaraj University, Madurai 625 021, India

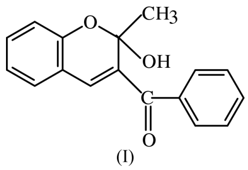
Correspondence e-mail: rafi@mrna.tn.nic.in

Received 2 January 2003
Accepted 27 January 2003
Online 7 February 2003

In the title compound, $C_{17}H_{14}O_3$, the conformation of the pyran ring is intermediate between sofa and half-chair. In the crystal structure, the hydroxyl and carbonyl O atoms are involved in O—H···O intermolecular hydrogen bonding, forming chains along the *a* axis.

Comment

Chromene derivatives possess photochromic properties, and some 2*H*-chromene derivatives are found to possess anti-microbial activities (El-Gaby *et al.*, 2000). They also exhibit antifeedant activity against larvae of Spilarctia obliqua (Agarwal *et al.*, 2000). Endothelin-A (ET_A) selective receptor's antagonists contain the 2*H*-chromene moiety (Ishizuka *et al.*, 2002). The photochromic nature and the ET_A receptor involvement of chromenes prompted our crystallographic study of these compounds.



(I)

In the title compound, (I) (Fig. 1), the pyran ring is in a conformation which is intermediate between sofa and half-chair, with asymmetry parameters $\Delta C_s(C2) = 0.057\,(2)$ and $\Delta C_2(C2—O1) = 0.035\,(1)$ (Nardelli, 1983). Atoms C2 and O1 deviate from the weighted least-squares plane through the remaining four atoms of the pyran by −0.348 (3) and 0.120 (3) Å, respectively. The hydroxyl and methyl groups at C2 adopt pseudo-axial and pseudo-equatorial positions, respectively. The conformation of the attachment of the benzoyl substituent to the pyran ring is best described by the torsion angle C4—C3—C13—O14 of −150.4 (3)°. The C3—C13—C15—C20 torsion angle [53.7 (4)°] shows how the benzoyl phenyl ring is oriented. The phenyl ring (C15—C20) makes a dihedral angle of 70.2 (1)° with the mean plane through atoms C3–C10.

As observed in other chromene derivatives (Aldoshin *et al.*, 1995, 1996), the valence angles at the sp^3-hybridized atom C2 show substantial distortion from the ideal tetrahedral angle (109.5°), with maximum distortions for angles O1—C2—C12 [103.4 (3)°] and C12—C2—C3 [116.5 (3)°]. The Csp^3—O and Csp^2—O distances [C2—O1 = 1.443 (4) Å and O1—C9 = 1.363 (4) Å] agree with the average values given by Allen *et al.* (1987) for such distances in tetrahydropyran and furan ring systems. The C3—C4 [1.350 (4) Å] bond length is slightly longer than the previously reported value of 1.312 (5) Å for a

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.050
wR factor = 0.171
Data-to-parameter ratio = 9.0

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

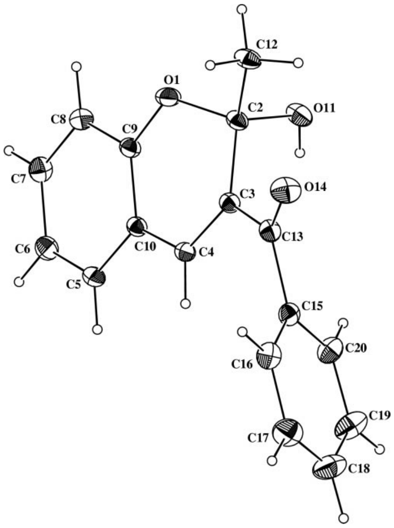


Figure 1
The structure of the title compound, showing 35% probability displacement ellipsoids and the atom-numbering scheme for the non-H atoms.

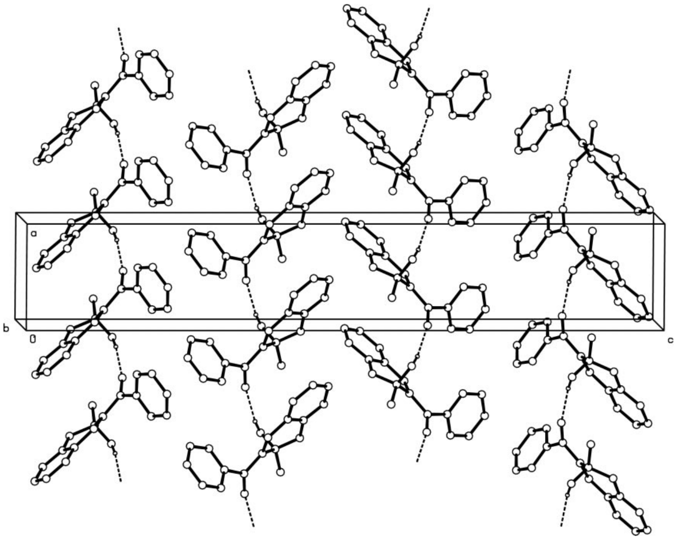


Figure 2
The molecular packing, viewed down the *b* axis.

diphenylchromene derivative (Aldoshin *et al.*, 1995) and 1.332 (4) Å in a dimethylchromene derivative (Aldoshin *et al.*, 1996).

The molecular structure of (I) is influenced by a weak C12—H12*A*···O14 interaction (Table 2). In the crystal structure, the molecules translated by one unit along the *a* axis are linked by O—H···O hydrogen bonds, involving the hydroxyl and carbonyl O atoms, to form molecular chains (Fig. 2).

Experimental

Benzoylacetone (10 mmol) in ethanol was mixed with salicylaldehyde (10 mmol), to which was added 0.5 ml of piperidine. The solution was stirred thoroughly for about 3 h at 273 K. This mixture was kept in a refrigerator for 12 h. A pale yellow product was obtained and this was separated out. This was filtered off and 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

$C_{17}H_{14}O_3$
$M_r = 266.28$
Orthorhombic, $P2_12_12_1$
$a = 5.691$ (4) Å
$b = 6.902$ (3) Å
$c = 34.044$ (2) Å
$V = 1337.2$ (11) Å^3
$Z = 4$
$D_x = 1.323$ Mg m^{-3}

Cu *Kα* radiation
Cell parameters from 25 reflections
$\theta = 15$–$35°$
$\mu = 0.73$ mm^{-1}
$T = 293$ (2) K
Prism, pale yellow
0.20 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: none
1645 measured reflections
1645 independent reflections
1563 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 74.9°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 42$
2 standard reflections every 60 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.171$
$S = 1.06$
1645 reflections
183 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0983P)^2 + 0.8035P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0103 (16)

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.363 (4)	C3—C4	1.350 (4)
O1—C2	1.443 (4)	C13—O14	1.215 (4)
C2—O11	1.405 (4)		
O11—C2—O1	108.8 (3)	O11—C2—C3	109.8 (3)
O11—C2—C12	107.3 (3)	O1—C2—C3	110.7 (2)
O1—C2—C12	103.4 (3)	C12—C2—C3	116.5 (3)
C4—C3—C13—O14	−150.4 (3)	C3—C13—C15—C20	53.7 (4)
C2—C3—C13—O14	30.6 (5)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12*A*···O14	0.96	2.55	2.884 (5)	100
O11—H11···O14^{i}	0.82	2.06	2.865 (4)	165

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

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 an O—H distance of 0.82 Å. The reflections (314), (208), (322) and (332) were omitted during the refinement as they fit very badly. Since Friedel pairs were not measured during the data collection, we were unable to determine the absolute configuration.

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

Acta Cryst. (2003). E**59**, o266–o268 M. Yogavel *et al.* • $C_{17}H_{14}O_3$ **o267**

(Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

ZAR thanks the Department of Biotechnology (DBT), Government of India, for a research grant.

References

Agarwal, S. K., Verma, S., Singh. S. S., Tripathi, A. K., Khan, Z. K. & Kumar, S. (2000). *J. Ethnopharmacol.* **71**, 231–234.

Aldoshin, S., Chuev, I., Philipenko, O., Pozzo, J.-L., Lokshin, V., Pépe, G. & Samat, A. (1996). *Acta Cryst.* C**52**, 1537–1539.

Aldoshin, S., Chuev, I., Utenyshev, A., Lokshin, V., Pozzo, J.-L., Pépe, G. & Guglielmetti, R. (1995). *Acta Cryst.* C**51**, 141–143.

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.

El-Gaby, M. S., Zahran, M. A., Ismail, M. M. & Ammar, Y. A. (2000). *Farmaco*, **55**, 227–232.

Enraf–Nonius (1994). *CAD*-4 *EXPRESS*. Enraf–Nonius, Delft, The Netherlands.

Harms, K. & Wocadlo, S. (1995). *XCAD*4. University of Marburg, Germany.

Ishizuka, N., Matsumura, K., Sakai, K., Fujimoto, M., Mihara, S. & Yamamori, T. (2002). *J. Med. Chem.* **45**, 2041–2055.

Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.

o268 M. Yogavel *et al.* • $C_{17}H_{14}O_3$ *Acta Cryst.* (2003). E**59**, o266–o268