Structure of 6-Hydroxyflavone†

J. SEETHARAMAN AND S. S. RAJAN*

*Department of Crystallography and Biophysics,
University of Madras, Madras-25, India*

Fig. 2. Stereoview of the unit-cell packing of the molecule viewed along the *a* axis.

Abstract

The benzopyran and phenyl rings are planar and the phenyl ring is rotated by 9.8 (2)° from the plane of the benzopyran ring. There is no correlation between the stretching of the C(2)—C(1′) bond and the relative orientation of the phenyl and benzopyran rings. The molecules in the unit cell are stabilized by van der Waals forces.

Comment

As part of our studies on the bioflavonoids and their analogues, the structure determination of the compound 6-hydroxyflavone was undertaken. In this structure, the γ-pyran ring, the benzene ring and the phenyl ring are almost planar ($\chi^2 = 31$, 7 and 4 respectively). The phenyl ring is rotated from the plane of the benzopyran ring through an angle of 9.8 (2)°. This is close to the value observed in 5-hydroxyflavone [5.7 (7)°] (Shoja, 1990). An analysis of this structure along with other different flavone derivatives (Vijayalakshmi, Rajan & Srinivasan, 1986, 1987; Wallet, Gaydou, Fadlane & Baldy, 1988; Wallet, Gaydou & Baldy, 1989; Shoja, 1989, 1990; Jin, Yamagata & Tomita, 1990; Wallet, Gaydou, Tinant, Declercq, Baldy & Bonifassi, 1990; Watson,
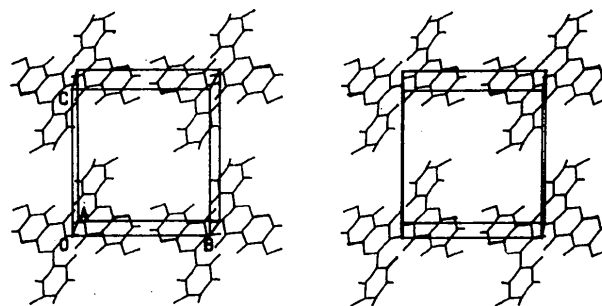
Kashyap, Gao & Mabry, 1991) shows that there is no correlation between the stretching of the C(2)—C(1′) bond and the relative orientation of the phenyl ring with respect to the benzopyran ring. It should be noted that all the flavones crystallize in centrosymmetric systems. A view of the molecule of 6-hydroxyflavone along the *b* axis with atom labelling is shown in Fig. 1 and a stereoview of the unit-cell packing of the molecule is shown in Fig. 2 (*PLUTO*, Motherwell, 1976). The molecules are stabilized by van der Waals forces. Diagrams and most calculations were performed on VAX 730 and MicroVAX II computers.

Experimental

Crystal data

$C_{15}H_{10}O_3$	$D_x = 1.416$ Mg m^{-3}
$M_r = 238$	$D_m = 1.411$ (3) Mg m^{-3}
Triclinic	Cu $K\alpha$ radiation
$P\bar{1}$	$\lambda = 1.5418$ Å
$a = 5.236$ (1) Å	Cell parameters from 20
$b = 10.246$ (2) Å	reflections
$c = 10.884$ (2) Å	$\theta = 20$–30°
$\alpha = 89.368$ (4)°	$\mu = 0.76$ mm^{-1}
$\beta = 76.092$ (3)°	$T = 293$ K
$\gamma = 86.412$ (4)°	Needle
$V = 565.7$ (3) Å^3	0.25 × 0.20 × 0.20 mm
$Z = 2$	Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.03$
$\omega/2\theta$ scans	$\theta_{max} = 60°$
Absorption correction: none	$h = 0 \rightarrow 6$
	$k = -11 \rightarrow 11$
1794 measured reflections	$l = -12 \rightarrow 12$
1794 independent reflections	3 standard reflections
1336 observed reflections	monitored every 200
$[I > 3\sigma(I)]$	reflections
	intensity variation: <2%

Refinement

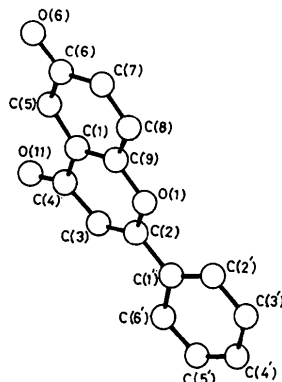|	
Refinement on F	$w = 1/[\sigma^2(F)+0.01939F^2]$
Final $R = 0.057$	$(\Delta/\sigma)_{max} = 0.00189$
$wR = 0.056$	$\Delta\rho_{max} = 0.3092$ e Å^{-3}
$S = 0.783$	$\Delta\rho_{min} = -0.2024$ e Å^{-3}

Fig. 1. Molecule of 6-hydroxyflavone viewed along the *b* axis with atom labelling.

1336 reflections
163 parameters
All H-atom parameters re-
   fined

Atomic scattering factors
   from *International Tables*
   for X-ray Crystallography
   (1974, Vol. IV)

Cell refinement and data reduction: *SDP* (Frenz, 1978). Program used to solve structure: *SHELX86* (Sheldrick, 1986). Programs used to refine structure: *SHELX76* (Sheldrick, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983). Refinement was by full-matrix least-squares methods. The density of the crystal was measured by flotation.

We thank Professor R. Srinivasan for discussions and one of us (JS) thanks UGC (India) for financial assistance.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* ($\mathring{A}^2$)

$$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	*y*	*z*	U_{eq}
O(1)	0.2738 (9)	0.2641 (4)	0.1781 (4)	0.515 (2)
C(2)	0.1111 (14)	0.1672 (6)	0.1831 (6)	0.532 (3)
C(3)	0.1414 (14)	0.0815 (6)	0.0880 (6)	0.506 (3)
C(4)	0.3381 (12)	0.0881 (5)	−0.0272 (5)	0.435 (3)
C(5)	0.7320 (14)	0.2104 (6)	−0.1319 (6)	0.523 (3)
C(6)	0.9032 (12)	0.3082 (5)	−0.1331 (5)	0.456 (3)
O(6)	1.0972 (10)	0.3255 (4)	−0.2345 (4)	0.390 (3)
C(7)	0.8593 (14)	0.3898 (6)	−0.0253 (6)	0.521 (3)
C(8)	0.6550 (13)	0.3740 (5)	0.0754 (6)	0.492 (3)
C(9)	0.4805 (13)	0.2759 (5)	0.0727 (6)	0.444 (3)
C(10)	0.5180 (12)	0.1938 (5)	−0.0290 (5)	0.414 (3)
O(11)	0.3621 (10)	0.0118 (4)	−0.1155 (4)	0.611 (3)
C(1′)	−0.0910 (14)	0.1694 (5)	0.3041 (5)	0.480 (3)
C(2′)	−0.0730 (15)	0.2471 (6)	0.4031 (6)	0.585 (4)
C(3′)	−0.2636 (17)	0.2465 (7)	0.5162 (7)	0.670 (5)
C(4′)	−0.4799 (18)	0.1676 (8)	0.5299 (8)	0.731 (5)
C(5′)	−0.4952 (15)	0.0916 (7)	0.4333 (8)	0.658 (4)
C(6′)	−0.3114 (15)	0.0916 (7)	0.3210 (6)	0.639 (4)

Table 2. *Geometric parameters* ($\mathring{A}$, °)

O(1)—C(2)	1.341 (8)	C(6)—C(7)	1.413 (8)
O(1)—C(9)	1.385 (7)	C(7)—C(8)	1.351 (8)
C(2)—C(3)	1.338 (9)	C(8)—C(9)	1.405 (9)
C(2)—C(1′)	1.477 (8)	C(9)—C(10)	1.366 (8)
C(3)—C(4)	1.423 (8)	C(1′)—C(2′)	1.371 (9)
C(4)—C(10)	1.476 (8)	C(1′)—C(6′)	1.418 (10)
C(4)—O(11)	1.223 (7)	C(2′)—C(3′)	1.386 (9)
C(5)—C(6)	1.384 (9)	C(3′)—C(4′)	1.410 (13)
C(5)—C(10)	1.398 (8)	C(4′)—C(5′)	1.336 (12)
C(6)—O(6)	1.327 (7)	C(5′)—C(6′)	1.362 (10)
C(2)—O(1)—C(9)	119.6 (5)	O(1)—C(9)—C(8)	117.4 (5)
O(1)—C(2)—C(1′)	111.2 (5)	C(8)—C(9)—C(10)	120.9 (6)
O(1)—C(2)—C(3)	121.7 (6)	O(1)—C(9)—C(10)	121.6 (6)
C(3)—C(2)—C(1′)	127.1 (6)	C(5)—C(10)—C(9)	118.7 (6)
C(2)—C(3)—C(4)	123.6 (6)	C(4)—C(10)—C(9)	119.9 (5)
C(3)—C(4)—O(11)	123.7 (6)	C(4)—C(10)—C(5)	121.4 (5)
C(3)—C(4)—C(10)	113.5 (5)	C(2)—C(1′)—C(6′)	120.8 (5)
C(10)—C(4)—O(11)	122.7 (5)	C(2)—C(1′)—C(2′)	121.3 (6)
C(6)—C(5)—C(10)	121.5 (6)	C(2′)—C(1′)—C(6′)	117.9 (6)
C(5)—C(6)—C(7)	117.9 (6)	C(1′)—C(2′)—C(3′)	120.3 (7)
C(5)—C(6)—O(6)	120.2 (5)	C(2′)—C(3′)—C(4′)	120.4 (7)
O(6)—C(6)—C(7)	121.9 (5)	C(3′)—C(4′)—C(5′)	119.0 (8)
C(6)—C(7)—C(8)	121.2 (6)	C(4′)—C(5′)—C(6′)	121.6 (8)
C(7)—C(8)—C(9)	119.7 (6)	C(1′)—C(6′)—C(5′)	120.9 (7)

References

Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution*. In *Computing in Crystallogrpahy*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft Univ. Press.
Jin, G.-Z., Yamagata, Y. & Tomita, K.-I. (1990). *Acta Cryst.* C46, 310–313.
Motherwell, W. D. S. (1976). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
Nardelli, M. (1983). *Comput. Chem.* 7, 95–98.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
Sheldrick, G. M. (1986). *SHELX86*. Program for crystal structure determination. Univ. of Göttingen, Germany.
Shoja, M. (1989). *Acta Cryst.* C45, 828–829.
Shoja, M. (1990). *Acta Cryst.* C46, 517–519.
Vijayalakshmi, J., Rajan, S. S. & Srinivasan, R. (1986). *Acta Cryst.* C42, 1752–1754.
Vijayalakshmi, J., Rajan, S. S. & Srinivasan, R. (1987). *Acta Cryst.* C43, 1998–2020.
Wallet, J. C., Gaydou, E. M. & Baldy, A. (1989). *Acta Cryst.* C45, 512–515.
Wallet, J. C., Gaydou, E. M., Fadlane, A. & Baldy, A. (1988). *Acta Cryst.* C44, 357–359.
Wallet, J. C., Gaydou, E. M., Tinant, B., Declercq, J.-P., Baldy, A. & Bonifassi, P. (1990). *Acta Cryst.* C46, 1131–1133.
Watson, W. H., Kashyap, R. R., Gao, F. & Mabry, T. J. (1991). *Acta Cryst.* C47, 459–461.

1,4-Dibenzoylbenzene

Tsonko Kolev, Hans Preut and Paul Bleckmann

Fachbereich Chemie, Universität Dortmund, Postfach 500500, D-4600 Dortmund 50, Germany

Ivan Juchnovski

Institute for Organic Chemistry, Bulgarian Academy of Sciences, Sofia 1040, Bulgaria

Abstract

The structure determination of the title compound was undertaken in order to obtain more structural information about phthalic acid derivatives and the effects of different benzoyl group positions on the conformation of these compounds. The benzoyl fragments of the centrosymmetric molecule are coplanar and form an angle of 121.4(1)° with the central phenylene ring. The bond angles around the C atom of the carbonyl group do not deviate significantly from 120°, indicating sp^2 hybridization. The torsion angles C(7)—C(6)—C(4)—O(5) and C(2)—C(3)—C(4)—O(5) are 23.5(5) and 36.9(5)°, respectively; bond distances and angles are within the normal ranges.