

Fig. 2. The crystal structure projected along the b axis. Hydrogen bonds are shown as narrow lines.

$<0.7\%$; 2580 reflections measured; 1788 observed reflections with $F_o^2 > \sigma(F_o^2)$. Systematic absences $0k0$, k odd. No corrections for absorption. Structure solved by direct methods with *MULTAN* (Main, Woolfson & Germain, 1971). Refined by full-matrix least squares. The locations of 45 H atoms were found on difference Fourier maps. Non-H atoms refined with anisotropic thermal parameters, H atoms with fixed isotropic thermal parameters ($B = 5.0 \text{ \AA}^2$). $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1.0$ for $|F_o| < 81.84$, $w = (81.84/F_o)^2$ for $|F_o| \geq 81.84$. Final $R = 0.038$, $wR = 0.036$, $S = 2.40$ for 497 variables, secondary-extinction factor $g = 1.89(6) \times 10^{-6}$ [$|F_o| = |F_c|/(1 + g|c|)$]; $\Delta/\sigma < 0.23$, largest peak in final ΔF map $+0.14 \text{ e \AA}^{-3}$; atomic scattering factors from *International Tables for X-ray Crystallography*

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5-Hydroxyflavone*

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Abstract. $C_{15}H_{10}O_3$, $M_r = 238.24$, monoclinic, $P2_1/c$, $a = 4.746(1)$, $b = 18.758(3)$, $c = 12.944(2) \text{ \AA}$, $\beta = 95.38(2)^\circ$, $V = 1147.2(5) \text{ \AA}^3$, $Z = 4$, $D_x = 1.384 \text{ g cm}^{-3}$, $Cu K\alpha$ radiation, $\lambda = 1.5418 \text{ \AA}$, $\mu = 8.0 \text{ cm}^{-1}$, $F(000) = 496$, $T = 293 \text{ K}$. Final $R = 0.054$ for 1003 observed reflections. The 5-hydroxy group forms a cyclic intramolecular hydrogen bond with

(1974); programs: Enraf–Nonius *SDP* (Frenz, 1984), *ORTEPII* (Johnson, 1976). The structure of the title compound is shown in Fig. 1, crystal packing in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1, bond distances and angles are listed in Table 2.*

Related literature. Funiculosin is an antiviral and antifungal antibiotic (Ando, Suzuki, Saeki, Tamura & Arima, 1969), and its structure elucidation and antifungal activity have been reported (Ando, Matsuura, Nawata, Endo, Sasaki, Okutomi, Saeki & Tamura, 1978).

* Lists of anisotropic thermal parameters, H-atom coordinates, torsion angles, least-squares planes and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52364 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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the carbonyl group. The heterocyclic ring is coplanar with the benzene ring. The phenyl ring is slightly twisted, and makes a dihedral angle of $5.7(7)^\circ$ with the γ -benzopyrone portion of the molecule. The torsion angle for $C(2')-C(1')-C(2)-O(1)$ is $-5.8(4)^\circ$.

Experimental. The title compound (Fig. 1) was purchased from the Indofine Chemical Company.

* Flavone is 2-phenyl-4H-benzopyran-4-one.

Table 1. Final fractional coordinates and equivalent isotropic temperature factors for non-H atoms, with *e.s.d.'s* in parentheses

$$B_{eq} = \frac{4}{3} \sum_i \beta_j a_i \cdot a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
O(1)	0.6905 (4)	0.1751 (1)	0.2135 (1)	4.50 (4)
O(2)	0.2873 (5)	0.1164 (1)	-0.0645 (2)	6.73 (6)
O(3)	-0.0290 (5)	0.2291 (1)	-0.0604 (2)	6.38 (6)
C(1')	0.9897 (6)	0.0756 (1)	0.2289 (2)	4.27 (6)
C(2)	0.7645 (6)	0.1138 (1)	0.1665 (2)	4.20 (6)
C(2')	1.1207 (7)	0.1054 (2)	0.3195 (2)	5.41 (7)
C(3)	0.6347 (6)	0.0938 (1)	0.0749 (2)	4.92 (7)
C(3')	1.3339 (7)	0.0690 (2)	0.3769 (3)	6.78 (9)
C(4)	0.4134 (6)	0.1350 (2)	0.0206 (2)	4.79 (7)
C(4')	1.4223 (8)	0.0034 (2)	0.3461 (3)	6.72 (9)
C(5)	0.1184 (6)	0.2452 (2)	0.0316 (2)	4.87 (7)
C(5')	1.2964 (7)	-0.0266 (2)	0.2558 (3)	6.36 (8)
C(6)	0.0495 (7)	0.3054 (2)	0.0849 (2)	5.44 (7)
C(6')	1.0810 (7)	0.0089 (2)	0.1979 (2)	5.59 (7)
C(7)	0.1986 (6)	0.3207 (2)	0.1795 (2)	5.40 (7)
C(8)	0.4114 (6)	0.2771 (2)	0.2234 (2)	5.03 (7)
C(9)	0.4769 (6)	0.2175 (1)	0.1680 (2)	4.24 (6)
C(10)	0.3356 (6)	0.1996 (1)	0.0731 (2)	4.19 (6)

Table 2. Bond lengths (\AA), angles ($^\circ$) and selected torsion angles ($^\circ$), with *e.s.d.'s* in parentheses

O(1)—C(2)	1.362 (3)	C(3')—C(4)	1.372 (5)
O(1)—C(9)	1.376 (3)	C(4)—C(10)	1.452 (4)
O(2)—C(4)	1.253 (3)	C(4')—C(5')	1.381 (5)
O(3)—C(5)	1.358 (3)	C(5)—C(6)	1.378 (4)
C(1')—C(2)	1.465 (4)	C(5)—C(10)	1.406 (4)
C(1')—C(2')	1.392 (4)	C(5')—C(6')	1.381 (4)
C(1')—C(6')	1.395 (4)	C(6)—C(7)	1.386 (4)
C(2)—C(3)	1.338 (4)	C(7)—C(8)	1.378 (4)
C(3)—C(3')	1.378 (5)	C(8)—C(9)	1.379 (4)
C(3)—C(4)	1.433 (4)	C(9)—C(10)	1.385 (4)

C(2)—O(1)—C(9)	120.5 (2)	O(3)—C(5)—C(6)	119.6 (3)
C(2)—C(1')—C(2')	120.9 (2)	O(3)—C(5)—C(10)	119.9 (2)
C(2)—C(1')—C(6')	120.7 (2)	C(6)—C(5)—C(10)	120.5 (2)
C(2')—C(1')—C(6')	118.4 (3)	C(4')—C(5')—C(6')	120.2 (3)
O(1)—C(2)—C(1')	111.8 (2)	C(5)—C(6)—C(7)	119.1 (3)
O(1)—C(2)—C(3)	121.1 (2)	C(1')—C(2)—C(3)	120.7 (3)
C(1')—C(2)—C(3)	127.1 (2)	C(6)—C(7)—C(8)	122.3 (3)
C(1')—C(2')—C(3')	120.2 (3)	C(7)—C(8)—C(9)	117.3 (3)
C(2)—C(3)—C(4)	122.6 (3)	O(1)—C(9)—C(8)	116.1 (2)
C(2')—C(3')—C(4')	121.1 (3)	O(1)—C(9)—C(10)	121.0 (2)
O(2)—C(4)—C(3)	123.2 (3)	C(8)—C(9)—C(10)	122.9 (2)
O(2)—C(4)—C(10)	121.5 (3)	C(4)—C(10)—C(5)	122.5 (2)
C(3)—C(4)—C(10)	115.31 (2)	C(4)—C(10)—C(9)	119.6 (2)
C(3')—C(4')—C(5')	119.5 (3)	C(5)—C(10)—C(9)	117.8 (2)

O(1)—C(2)—C(3)—C(4)	0.3 (4)	C(2)—C(3)—C(4)—C(10)	-1.1 (4)
C(3)—C(4)—C(10)—C(5)	-178.6 (3)	C(3)—C(4)—C(10)—C(9)	0.9 (4)
C(4)—C(10)—C(9)—O(1)	0.0 (5)	C(4)—C(10)—C(5)—C(6)	179.2 (3)
C(10)—C(9)—O(1)—C(2)	-1.0 (4)	C(10)—C(5)—C(6)—C(7)	0.4 (4)
C(5)—C(6)—C(7)—C(8)	-1.0 (4)	C(6)—C(7)—C(8)—C(9)	1.4 (4)
C(7)—C(8)—C(9)—C(10)	-1.4 (4)	C(7)—C(8)—C(9)—O(1)	179.8 (3)
C(8)—C(9)—C(10)—C(4)	-178.7 (3)	C(8)—C(9)—C(10)—C(5)	0.9 (4)

Yellow needles were grown from a mixture of acetonitrile and methanol solution. Data were collected on an Enraf-Nonius CAD-4 diffractometer, graphite monochromator. The crystal had dimensions $0.15 \times 0.20 \times 0.40$ mm. Cell parameters were measured on the diffractometer using 25 reflections in the 2θ range $20-40^\circ$. Range of indices $-5 \leq h \leq 5$, $0 \leq k \leq 21$, $0 \leq l \leq 14$ ($\theta \leq 60^\circ$). Three standards, 011, $\bar{1}12$, 110, measured after every 200 reflections,

showed a variation of 0.1%. No absorption corrections. Lorentz and polarization corrections. 1703 unique reflections were measured. 1003 observed reflections with $|F_o| > 3\sigma(|F_c|)$. Direct methods (MULTAN82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) used for structure determination. H atoms located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. $\sum w(|F_o| - |F_c|)^2$ minimized. $w = 4F_o^2/S^2(I)$ with $S(I) = [\sigma^2(I) + p(F_o)^2]^{1/2}$, $p = 0.04$. $wR = 0.054$, max. $\Delta/\sigma = 0.02$. Max. peak height in the final difference Fourier map = 0.28 e \AA^{-3} , $S = 1.649$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Enraf-Nonius SDP (Frenz, 1984). Atomic parameters are given in Table 1; * the bond distances, bond angles and relevant torsion angles are presented in Table 2. Atomic numbering is shown in Fig. 1 and Fig. 2 is a packing diagram.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52330 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

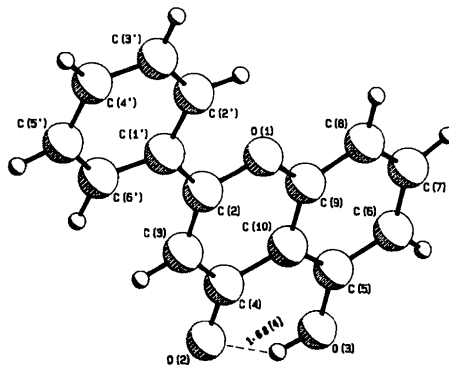


Fig. 1. Numbering of atoms and conformation of the molecule. The intramolecular hydrogen bond [$1.68(4) \text{ \AA}$] is shown as a dashed line.

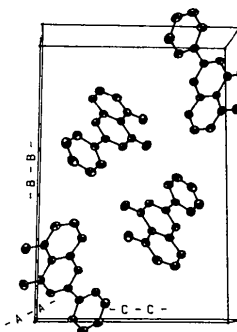


Fig. 2. Molecular packing diagram.

Related literature. In the generally preferred conformation the dihedral angle between the phenyl and γ -benzopyrone ring is expected to be small as shown in the case of the title compound where the value is $5.2(9)^\circ$. In related flavones, the reported increase in dihedral angle to 24.8 (Shoja, 1989) and 28° (Ting & Watson, 1972) has the effect of increasing the length of the C(1')—C(2) bond from $1.465(4)$ (Table 2), to $1.485(5)$ and $1.504(9)$ Å, respectively. Only intramolecular hydrogen bonding was found in the title compound in contrast to 3-hydroxyflavone which displayed both intra- and intermolecular hydrogen bonding (Etter, Urbanczyk-Lipkowska, Baer & Barbara, 1986).

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Structure of 3-Chloroflavanone

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Abstract. 3-Chloro-2-phenylbenzo- γ -pyrone, $C_{15}H_{11}ClO_2$, $M_r = 258.7$, monoclinic, $P2_1/n$, $a = 9.317(3)$, $b = 14.991(2)$, $c = 8.848(4)$ Å, $\beta = 94.45(3)^\circ$, $V = 1232.1$ Å³, $Z = 4$, $D_m = 1.37$, $D_x = 1.39$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 3.0$ cm⁻¹, $F(000) = 536$, $T = 295$ K, $R = 0.054$ for 2063 observed reflections ($|F_o| > 1\sigma|F_o|$). The pyran ring is puckered due to the saturation of the C(2)—C(3) bond. The puckering causes the pyran ring to be distorted into a C(2)-sofa conformation. Owing to the C(2)—C(3) saturation, there are two asymmetric centers present, one at C(2), the other at C(3). The exocyclic phenyl substituent is equatorially oriented at C(2) and the Cl(3) is axially oriented at C(3). The 2-phenyl substituent is twisted out of the benzo- γ -pyrone ring plane due to steric interactions with Cl(3) at C(3). The C(3)—C(2)—C(11)—C(12) torsion angle is $74.26(37)^\circ$.

Experimental. A white prism crystal of 3-chloroflavanone ($C_{15}H_{11}ClO_2$) was obtained by sublimation *in vacuo*. The crystal, with approximate dimensions $0.50 \times 0.25 \times 0.25$ mm, was mounted on a glass fiber. The density D_m was measured by flotation in a 38 wt% solution of calcium chloride. Preliminary Weissenberg film studies were performed on an Enraf-Nonius Diffractis 601 using Cu $K\alpha$ radiation ($\lambda = 1.54178$ Å). The preliminary film work was used to facilitate space-group assignment through

systematic extinctions. The crystal was mounted on an Enraf-Nonius CAD-4 computer-controlled κ -axis diffractometer with the long axis of the crystal parallel to the φ axis of the goniometer. Preliminary examination and data collection were performed with Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å) and a graphite-crystal incident-beam monochromator. Cell constants and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 25 reflections in the range $5 < \theta < 12^\circ$ measured by the computer-controlled diagonal slit method of centering. The data were collected at a temperature of 296(1) K, using the θ - 2θ scan technique. The scan rate varied from 2 to 7° min⁻¹ in ω . The variable scan rate allows rapid data collection for intense reflections, where a fast scan rate is used, and ensures good counting statistics for weak reflections, where a slow scan rate is used. Three standard reflections ($3\bar{3}2$, $1\bar{2}4$, $3\bar{5}0$) were remeasured every 4 h; no significant loss of these intensities was observed throughout data collection. A crystal orientation check was also made after every 240 reflections. 4518 independent reflections were collected with 2θ up to 50.0° in $h - 11/11$, $k - 17/0$, $l - 10/10$. Corrections for Lorentz and polarization effects were applied to the intensity data, while no absorption correction was carried out. The structure was solved by direct methods. The non-hydrogen atoms were located from difference Fourier synthesis