Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 5,3′-Dihydroxy-3,7,4′,5′-tetramethoxyflavone

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.052
*wR* factor = 0.160
Data-to-parameter ratio = 20.8

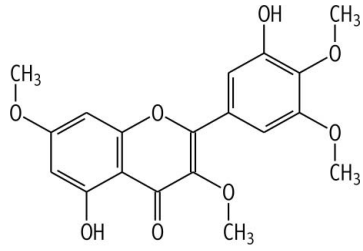
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 8 February 2005
Accepted 25 February 2005
Online 4 March 2005

The title compound, $C_{19}H_{18}O_8$ [systematic name: 5-hydroxy-2-(3-hydroxy-4,5-dimethoxyphenyl)-3,7-dimethoxychromen-4-one], an extract from *Premna tomentosa willd*, is used in the Siddha system of medicine practiced in South India. The dihedral angle between the mean planes through the benzopyran moiety and benzene ring is 38.60 (7)°. A strong O—H···O intramolecular hydrogen bond is observed in the molecular structure. O—H···O and C—H···O hydrogen bonds and π–π interactions stabilize the molecules in the crystal packing.

## Comment

*Premna tomentosa willd* (verbenaceae) (PT), commonly called 'Krishnapalai' or 'Pudangai Nari' in Tamil, is a valuable medicinal plant (Balakrishna *et al.*, 2003) used for the treatment of various disorders, such as hepatic disorders (through oral administration), stomach disorders and diarrhoea (Shanmugavelu, 1987). The methanol extract of the PT leaves affords protection against acetaminophen-induced hepatotoxicity in rats (Devi & Devaki, 1998), by its antioxidant property (Devi *et al.*, 1998). The anti-inflammatory effect of the plant has been reported by Alam *et al.*, 1993). In view of the above important properties, the plant extract was studied and a number of compounds have been derived. The title compound, (I), is one among the series, and here we report the crystal structure of (I). This compound can also be extracted from the leaves of *Citrus monspeliensis* (Berti *et al.*, 1967).




(I)

The benzopyran moiety is planar and the benzene ring attached at the C2 position is oriented at angle of 38.60 (7)° (Fig. 1). The methoxy group at C7 is coplanar with the benzopyran ring [C6—C7—O4—C12 = 1.2 (3)°], whereas that at C3 is twisted away from it [C11—O5—C3—C4 = −61.1 (3)°]. One of the methoxy groups attached to the C13—C18 benzene ring is almost coplanar with that ring [C19—O6—C15—C14 = 4.5 (3)°], whereas the other is rotated about the O7—C16 bond [C20—O7—C16—C15 = −76.8 (3)°]. The bond lengths and angles in (I) are comparable with those

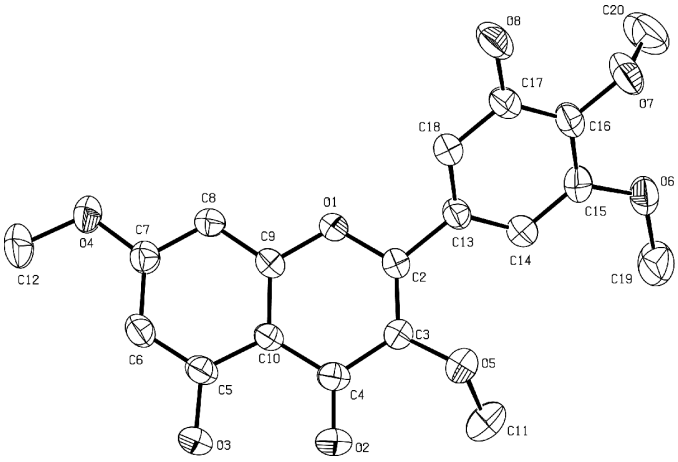



**Figure 1**
*PLATON* plot (Spek, 2003) of (I), showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

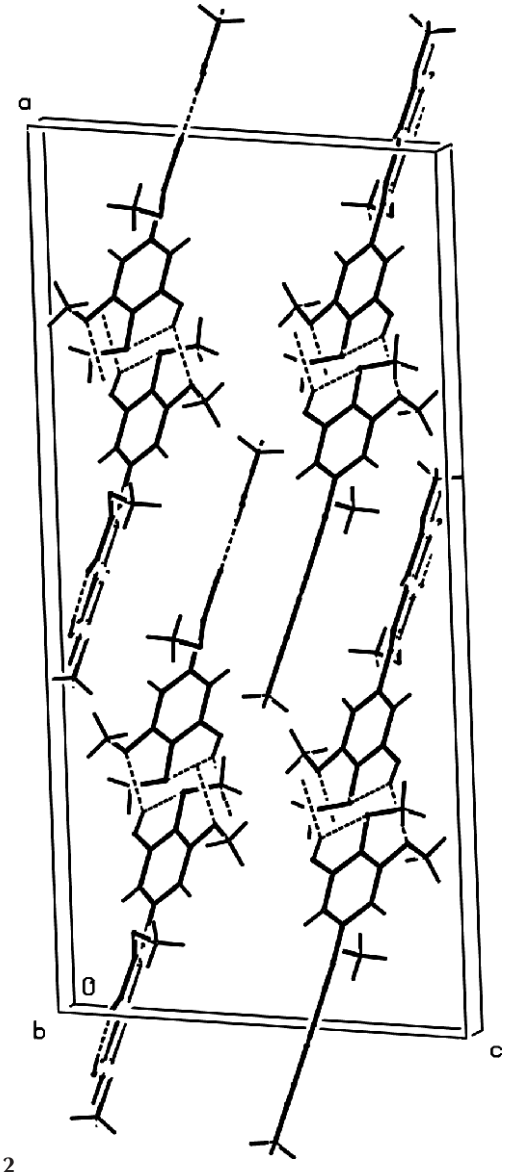



**Figure 2**
The packing of the molecules, viewed down the *b* axis. Dashed lines represent selected hydrogen bonds.

observed in 5-hydroxy-6,7,8,3′,4′,5′-hexamethoxyflavone (Vijayalakshmi *et al.*, 1987).

The OH group at the C5 position is strongly hydrogen bonded to the nearby carbonyl atom O2 and the other OH group at C17 is involved in an O8—H8*A*···O7 interaction (Table 1). The crystal packing is stabilized by both O—H···O and C—H···O intermolecular hydrogen bonds (Table 1). Along the *b* axis, the molecules form C8—H8···O2($x, 1 + y, z$) hydrogen-bonded chains (Fig. 2). Along the *c* axis, the benzopyran moieties of centrosymmetrically related molecules are stacked such that the distance between the centroids of the pyrone and benzene rings is 3.452 (2) Å, indicating significant $\pi$–$\pi$ interaction.

## Experimental

Compound (I) was isolated from the benzene-soluble part of the alcoholic extract of the leaves of the plant *Premna tomentosa willd* (family Verbenaceae). The benzene-soluble part was chromatographed over silica gel (100–200 mesh) and eluted with benzene–ethyl acetate (9:1) to give (I). The compound was crystallized from a hexane–benzene (1:1) solvent system (m.p. 423 K).

*Crystal data*

$C_{19}H_{18}O_8$
$M_r$ = 374.33
Monoclinic, $C2/c$
$a$ = 30.958 (7) Å
$b$ = 7.9544 (19) Å
$c$ = 14.263 (6) Å
$\beta$ = 95.53 (3)°
$V$ = 3495.9 (18) Å$^3$
$Z$ = 8
$D_x$ = 1.422 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 5092 reflections
$\theta$ = 1.3–30.0°
$\mu$ = 0.11 mm$^{-1}$
$T$ = 293 (2) K
Block, yellow
0.25 × 0.22 × 0.20 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: none
10241 measured reflections
5092 independent reflections
2382 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.061
$\theta_{max}$ = 30.0°
$h = -43 \rightarrow 43$
$k = -11 \rightarrow 11$
$l = -20 \rightarrow 0$
3 standard reflections
every 200 reflections
intensity decay: 1%

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.160
$S$ = 0.99
5092 reflections
245 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0772P)^2 + 0.2525P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0018 (3)

**Table 1**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O2 | 0.82 | 1.89 | 2.609 (2) | 146 |
| O8—H8*A*···O7 | 0.82 | 2.34 | 2.778 (3) | 114 |
| O8—H8*A*···O6$^{i}$ | 0.82 | 2.20 | 2.904 (3) | 144 |
| C8—H8···O2$^{ii}$ | 0.93 | 2.40 | 3.265 (3) | 155 |
| C11—H11*B*···O2 | 0.96 | 2.34 | 2.967 (4) | 122 |
| C14—H14···O5 | 0.93 | 2.54 | 2.936 (3) | 106 |
| C20—H20*B*···O6 | 0.96 | 2.52 | 3.064 (4) | 116 |

Symmetry codes: (i) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $x, 1 + y, z$.

All H atoms were positioned geometrically (O—H = 0.82 Å and C—H = 0.93 or 0.96 Å) and allowed to ride on their parent atoms, with $U_{iso}$(H) = 1.2 or 1.5 times $U_{eq}$(parent atom).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

## References


Alam, M., Joy, S., Susan, T. & Usman Ali, S. (1993). *Ancient Sci.* **13**, 185–188.

Balakrishna, K., Sukumar, E., Sarada, V. & Patra, A. (2003). *J. Indian Chem. Soc.* **80**, 792–793.

Berti, G., Livi, O., Segnini, D. & Cavero, I. (1967). *Tetrahedron*, **23**, 2295–2300.

Devi, K., Anandan, R., Devaki, T., Apparananthanm, T. & Balakrishna, K. (1998). *Biomed. Res.* **19**, 339–342.

Devi, K. & Devaki, T. (1998). *Med. Sci. Res.* **26**, 785–787.

Enraf–Nonius (1994). *CAD-4 EXPRESS*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.

Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.

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

Shanmugavelu, M. (1987). *Siddha Cure for Diseases*. Chennai: Tamilnadu Siddha Medical Board Publications.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Vijayalakshmi, J., Rajan, S. S. & Srinivasan, R. (1987). *Acta Cryst.* C**43**, 1998–2000.