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5,2′-Dihydroxy-7-methoxyflavanone

M. Krishnaiah,[a]* R. Ravi Kumar,[a] N. Jagadeesh Kumar,[a] D. Gunasekar[b] and B. Jayaprakasam[b]

[a]Department of Physics, Sri Venkateswara University, Tirupati (AP), India, and [b]Department of Chemistry, Sri Venkateswara University, Tirupati (AP), India

Correspondence e-mail: mkphysvu@yahoo.com

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.064
wR factor = 0.201
Data-to-parameter ratio = 12.3

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

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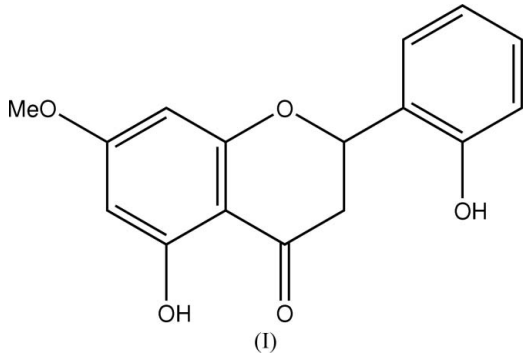
The γ-pyranone ring of the title molecule, $C_{16}H_{14}O_5$, adopts an envelope conformation. The crystal packing is stabilized by O—H···O and C—H···O hydrogen bonding.

Comment

The title compound, a flavanone also known as dihydroechioidinin, was isolated from the whole plant of *Andrographis echioides* Nees (Acanthaceae), an erect herb widely distributed in the dry districts of tropical India and Sri Lanka (Gamble, 1956). In order to study the biological activity of flavonoids, a number of flavone structures were determined, of which only a few have been reported to date (Cantrell *et al.*, 1974; Tomlin & Cantrell, 1990; Mariezcurrena, 1978; Kendi *et al.*, 1995*a*,*b*). Flavonones have recognized spasmolytic, expectorant, antiulcer, liver-protecting and antimicrobial properties (Gaber, 1986). The structure determination of the title compound, (I), was undertaken as a part of our ongoing structure–activity study aimed at designing more active compounds, and in order to study the conformational features of the compound.



(I)

The title compound is shown in Fig. 1 and selected geometric details are given in Table 1. The dihydropyranone ring adopts an envelope conformation; the deviation of atom C2 from the O1/C8a/C4a/C4/C3 plane is 0.537 (7) Å. A similar conformation is observed in 7-hydroxyflavonone, 7-ethoxycarbonyl methoxyflavanone (Kendi *et al.*, 1995*a*) and 7-hydroxy-4′-methoxyflavanone (Kendi *et al.*, 1995*b*). The dihedral angle between the O1/C3–C8/C4a/C8a plane and the C1′–C6′ benzene ring plane is 55.3 (1)°. The corresponding angles are 103.0 (1)° in 7-hydroxyflavanone, 121.5 (2)° in 7-ethoxycarbonylmethoxy flavanone (Kendi *et al.*, 1995*a*), 65.2 (1)° in 7-hydroxy-4′-methoxyflavanone (Kendi *et al.*, 1995*b*) and 70.8 (1)° in 5,7,4′-trimethoxyflavanone (Mariezcurrena, 1978).

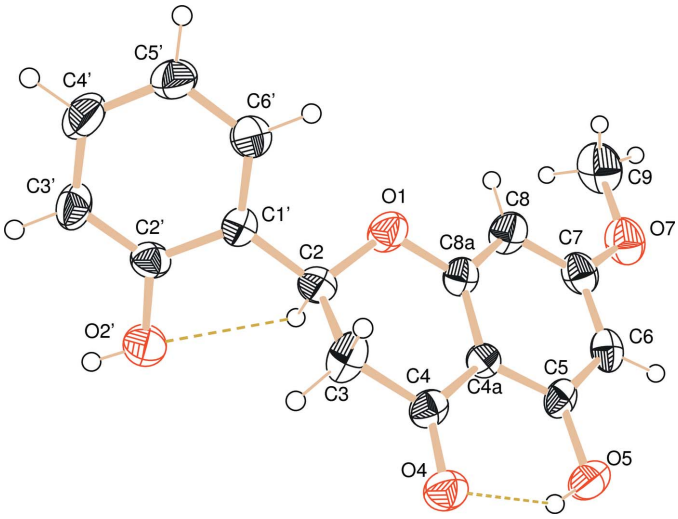


Figure 1
A view of the title compound, showing 40% probability displacement ellipsoids. Dashed lines indicate intramolecular hydrogen bonds.

The C2—C3 bond length of 1.500 (6) Å is in agreement with the corresponding values observed in flavanone structures: 1.502 (5) Å in 7-hydroxyflavanone (Kendi *et al.*, 1995*a*), 1.528 (7) Å in 7-hydroxy-4′-methoxyflavanone (Kendi *et al.*, 1995*b*) and 1.511 (5) Å in 3-chloroflavanone (Tomlin & Cantrell, 1990). In flavones, the C2—C3 bond is a double bond, with an average bond length of 1.34 Å (Kaneda *et al.*, 1973).

The crystal structure of (I) is stabilized by intermolecular O—H···O and C—H···O hydrogen bonds (Table 2).

Experimental

The dried and ground whole plant of *A. echioides* Nees (3 kg) was successively extracted with *n*-hexane (10 l), Me_2CO (10 l) and MeOH (10 l). The hexane extract, on purification over a silica-gel column using C_6H_6 as eluent, yielded 25 mg of the title compound (m.p. 473–474 K). Single crystals of (I) were obtained by slow evaporation of a chloroform solution.

Crystal data

$C_{16}H_{14}O_5$
M_r = 286.27
Monoclinic, $P2_1/c$
a = 10.1961 (10) Å
b = 7.5600 (8) Å
c = 18.243 (2) Å
β = 105.59 (2)°
V = 1354.5 (3) Å^3
Z = 4
D_x = 1.404 Mg m^{-3}
D_m = 1.40 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 2–25°
μ = 0.11 mm^{-1}
T = 298 (2) K
Needle, colourless
0.40 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.950, T_{max} = 0.970
2475 measured reflections
2346 independent reflections
1066 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 25.0°
$h = -12 \rightarrow 11$
$k = 0 \rightarrow 8$
$l = -20 \rightarrow 21$
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.201
S = 0.96
2346 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.096P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.402 (4)	C4—O4	1.248 (5)
C2—C1′	1.480 (5)	C5—O5	1.344 (4)
C2—C3	1.500 (6)	O7—C9	1.410 (5)
O1—C2—C3	113.5 (4)	C7—O7—C9	118.7 (4)
C2—C3—C4—C4a	25.9 (6)	O1—C2—C1′—C2′	149.4 (4)
C2—O1—C8a—C8	157.0 (4)	C3—C2—C1′—C6′	98.6 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2′—H2′···O4^{i}	0.82	1.94	2.752 (4)	170
O5—H5···O4	0.82	1.87	2.593 (4)	146
C2—H2···O2′	0.98	2.35	2.717 (5)	101
C5′—H5′···O5^{ii}	0.93	2.58	3.266 (6)	131

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

The H atoms were placed in idealized positions and constrained to ride on their parent atoms, with O—H = 0.82 Å and C—H = 0.93–0.97 Å, and with U_{iso}(H) = 1.2 or 1.5U_{eq}(carrier atom).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1994); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* in *WinGX* (Farrugia, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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