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5-Hydroxy-7,8,2′-trimethoxyflavone

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Molecules of the title compound, $C_{18}H_{16}O_6$, are almost planar. The dihedral angle between the benzopyran system and the benzene ring is 3.36 (5)°. In addition to an intramolecular O—H···O hydrogen bond, molecules are connected by weak intermolecular C—H···O hydrogen bonds to form a two-dimensional framework.

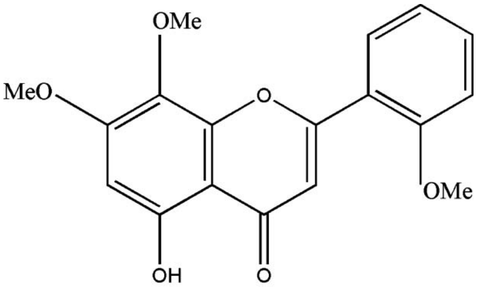
Key indicators

Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.094
Data-to-parameter ratio = 9.3

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

Comment

The title compound 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). The leaf juice of the plant is used as a remedy for fevers (Kirtikar & Basu, 1975). The study of the leaves leads to the isolation of the flavones, dihydroechioidinin, echioidinin, ethioidin, skullcapflavone 1,2′-*O* methyl ether and skullcapflavone 1,2′-*O* glucoside (Govindachari *et al.*, 1965; Jayaprakasam *et al.*, 1999). As a part of our ongoing investigation of medicinal plants, we report here the structure of the title ‘skullcapflavone 1,2′-*O* methyl ether’ (I), which was isolated by Jayaprakasam *et al.* (1999).



(I)

The title compound is shown in Fig. 1 and selected geometry is given in Table 1. The benzopyran ring is essentially planar, with a maximum deviation from the plane of 0.023 (1) Å. The benzene ring and the benzopyran group are almost coplanar, with a dihedral angle of 3.36 (5)° between their least-squares planes and an O1—C2—C9—C10 torsion angle of [176.22 (11) Å]. The C2—C9 bond length of 1.4733 (18) Å is comparable with that in 6-methylflavone [1.472 (2) Å], where the dihedral angle between the benzene ring and the benzopyran group is 8.9 (3)° (Hall *et al.*, 2001), flavone-3′-sulfonamide [1.478 (3) Å], where the dihedral angle is 8.2 (3)° (Kendi *et al.*, 2000), 5-hydroxyflavone [1.465 (4) Å], where the dihedral angle is 5.2 (9)° (Shoja, 1990), and 5,7-dihydroxy-4′-methoxy flavone [1.453 Å], where the corresponding angle is 3.1° (Shoja, 1992). Generally in flavones, the small dihedral angle results in a short C—C bond length between the

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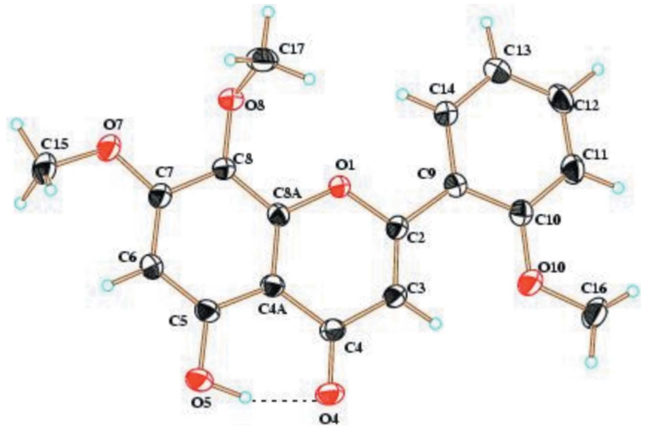

Figure 1
View of the title compound, showing 40% probability displacement ellipsoids. A dashed line indicates the intramolecular hydrogen bond.

benzene ring and the benzopyran group. On the other hand, flavone compounds that have a larger dihedral angle between the benzene ring and the benzopyran group have a significantly longer C—C distance for the bond joining the two moieties, *e.g.* 2′-methyl-3′-nitroflavone [139.8 (2)° and 1.491 (8) Å; Kendi *et al.*, 1996] and 5,4′-dihydroxy-3,6,7,8-tetramethoxyflavone [164.4 (6)° and 1.503 (8) Å; Vijayalakshmi *et al.*, 1986].

In addition to an intramolecular O—H⋯O hydrogen bond, molecules are linked *via* weak intermolecular C—H⋯O hydrogen bonds to form a two dimensional network (Table 2).

Experimental

The dried and ground whole plant of *A. echioides* Nees (3 kg) was successively extracted with *n*-hexane, Me_2CO and MeOH. The hexane extract, on purification over a silica gel column using C_6H_6 as eluant, yielded 100 mg of the sample.

Crystal data

$C_{18}H_{16}O_6$
$M_r = 328.31$
Triclinic, $P\bar{1}$
$a = 7.602$ (2) Å
$b = 8.7563$ (10) Å
$c = 11.642$ (3) Å
$\alpha = 85.252$ (16)°
$\beta = 84.838$ (12)°
$\gamma = 78.469$ (14)°
$V = 754.6$ (3) Å^3
$Z = 2$

$D_x = 1.445$ Mg m^{-3}
$D_m = 1.44$ Mg m^{-3}
D_m measured by KI flotation
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 2$–25°
$\mu = 0.11$ mm^{-1}
$T = 297$ (2) K
Rectangular **block?**, yellow
0.48 × 0.25 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.950$, $T_{\max} = 0.970$
2854 measured reflections
2635 independent reflections
2289 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.006$
$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 13$
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.094$
$S = 1.07$
2635 reflections
282 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 0.1561P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.121 (7)

Table 1
Selected geometric parameters (Å, °).

O4—C4	1.2519 (16)	C2—C9	1.4733 (18)
O5—C5	1.3542 (16)		
C8—O8—C17	113.37 (11)	C10—O10—C16	119.09 (12)
C7—O7—C15	119.10 (12)		
C4—C4*A*—C8*A*—C8	−178.29 (11)	C3—C2—C9—C10	3.7 (2)
O1—C2—C9—C14	2.86 (16)	O1—C2—C9—C10	−176.22 (11)

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O5—H5⋯O4	0.95 (2)	1.69 (2)	2.5855 (15)	156 (2)
C6—H6⋯O5$^{\text{i}}$	0.96 (2)	2.50 (2)	3.4250 (19)	162 (1)
C13—H13⋯O7$^{\text{ii}}$	0.96 (2)	2.54 (2)	3.497 (2)	174 (2)
C16—H16*C*⋯O4$^{\text{iii}}$	0.97 (2)	2.47 (3)	3.402 (2)	162 (2)

Symmetry codes: (i) $-x+1, -y, -z+2$; (ii) $-x, -y+1, -z+1$; (iii) $-x+1, -y-1, -z+1$.

All H atoms were clearly located in difference Fourier maps and refined independently with isotropic displacement parameters. The refined C—H distances range from 0.941 (16) to 1.01 (2) Å.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); 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: *ZORTEP* in *WinGX*; software used to prepare material for publication: *SHELXL97*.

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References

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Gamble, J. S. (1956). *Flora of the Presidency of Madras.* Vol. 2, p. 1051. Calcutta: Botanical Survey of India.

Govindachari, T. R., Parthasarathy, P. C., Pai, B. R. & Subramanyam, P. S. (1965). *Tetrahedron*, **21**, 2633, 3715.

Hall, B. J., Hanrahan, J. R., Janson, G. A. R., Hambley, T. W. & Hibbs, D. E. (2001). *Acta Cryst.* E**57**, o592–o593.

Jayaprakasam, B., Damu, A. G. & Gunasekhar, D. (1999). *Phytochemistry*, **52** 1–3.

Kendi, E., Ozbey, S., Bozdag, O. & Eartan, R. (1996). *Cryst. Res. Technol.* **5**, 611–615.

Kendi, E., Ozbey, S., Tuncbilek, M. & Eartan, R. (2000). *Acta Cryst.* C**56**, 457–458.

Kirtikar, K. R. & Basu, B. D. (1975). *Indian Medicinal Plants*, Vol. 3. NewDelhi: Periodical Experts.

North, A. C. T., Phillips, D. C & Mathews, F. S. (1968). *Acta Cryst*, A**24**, 351–359.

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

Shoja, M. (1990). *Acta Cryst.* C**46**, 517–519.

Shoja, M. (1992). *Acta Cryst.* C**48**, 2033–2035.

Vijayalakshmi, J., Rajan, S. S., Srinivasan, R. & Ramachandran Nair, A. G. (1986). *Acta Cryst.* C**42**, 1752–1753.