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5-Hydroxy-4′,6,7-trimethoxyflavone

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Key indicators

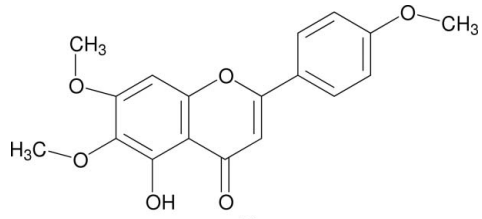
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.121
Data-to-parameter ratio = 12.1

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

The title compound, $C_{18}H_{16}O_6$, was isolated from *Salvia hypoleuca*. Except for one methoxy C atom, all non-H atoms are approximately coplanar. C—H···O hydrogen bonds generate a centrosymmetric $R_2^2(14)$ dimer. Inversion-related molecules are stacked along the b axis with significant π–π interactions.

Comment

The family Lamiaceae (Labiatae) is well known for its anti-tumor diterpenoidal constituents (Fujita & Node, 1984). The genus *Salvia* is the largest genus of this family, comprising over 800 species. The allelochemical potential of the title compound, (I), has been reported (Anaya *et al.*, 2003). The crystal structure of (I) has been previously reported by Kanko *et al.* (2004) [Cambridge Structural Database (CSD; Allen 2002) refcode GAJBAV] with an R value of 0.073; however, no coordinates are available for (I) in the CSD.



(I)

All bond lengths in (I) show normal values (Allen *et al.*, 1987). The benzopyran ring system, the C1′–C6′ benzene ring and the methoxy groups attached at C7 and C4′ are approximately coplanar (Fig. 1). The methoxy group attached at C6 is twisted away from this plane, with a C12—O4—C6—C7 torsion angle of 80.99 (16)°. The short H5′*A*···H7′*B* (2.20 Å) and H8*A*···H11*C* (2.25 Å) contacts cause widening of the O5—C7—C8 [123.77 (12)°] and O4′—C4′—C5′ [124.58 (12)°] bond angles from 120°.

Intramolecular O3—H3*A*···O2 and C2′—H2′*A*···O1 hydrogen bonds generate graph-set motifs $S(5)$ and $S(6)$, respectively (Bernstein *et al.*, 1995). Intermolecular C—H···O hydrogen bonds (Table 2) link the molecules into centrosymmetric $R_2^2(14)$ dimers (Fig. 2). Inversion-related molecules are stacked along the b axis in such a way that the centroid–centroid distance between the pyran and C1′–C6′ benzene rings is 3.6190 (8) Å (symmetry code: $1-x, 1-y, 1-z$).

Experimental

Salvia hypoleuca was collected in Iran (Kamdovan) in July 1999 and identified by Professor Sanei Chariat Pannahi, Karaj Agriculture

College, University of Tehran, Iran, where the voucher specimen of the collected material was deposited in the herbarium. The dried plant material (3 kg) was soaked in petroleum ether for a period of 7 d. The dark-coloured extract was filtered and evaporated at low temperature (303 K) and at low pressure (300 mbar) to yield a gummy mass. This gummy mass was chromatographed on a silica-gel column with increasing polarities of petroleum ether, petroleum ether–chloroform, chloroform, chloroform–methanol and, finally, methanol as mobile phase. Elution with 10% $CHCl_3$ in petroleum ether resulted in the yellow crystalline compound (I) (10.7 mg, m.p. 468 K).

Crystal data

$C_{18}H_{16}O_6$
$M_r = 328.31$
Triclinic, $P\overline{1}$
$a = 7.0968$ (3) Å
$b = 7.4959$ (3) Å
$c = 14.9563$ (6) Å
$\alpha = 76.681$ (1)°
$\beta = 85.757$ (1)°
$\gamma = 81.381$ (1)°
$V = 764.84$ (5) Å^3
$Z = 2$
$D_x = 1.426$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5177 reflections
$\theta = 2.8$–28.1°
$\mu = 0.11$ mm^{-1}
$T = 293$ (2) K
Slab, yellow
0.61 × 0.32 × 0.19 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.937$, $T_{max} = 0.980$
7381 measured reflections
2677 independent reflections
2416 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 25.0°$
$h = -8 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.121$
$S = 1.00$
2677 reflections
221 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0775P)^2 + 0.12P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.3613 (15)	O4—C6	1.3792 (14)
O1—C9	1.3707 (15)	O4—C12	1.4149 (18)
O2—C4	1.2558 (15)	O4′—C4′	1.3594 (16)
O3—C5	1.3498 (15)	O4′—C7′	1.4236 (18)
O5—C7	1.3567 (15)	C2—C3	1.3523 (18)
O5—C11	1.4262 (17)		
C7—O5—C11	117.62 (10)	C4′—O4′—C7′	117.55 (12)
C6—O4—C12	114.58 (10)		
C12—O4—C6—C7	80.99 (16)	C7′—O4′—C4′—C5′	−5.1 (2)
C11—O5—C7—C8	−5.84 (19)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O2	0.82	1.84	2.5737 (15)	148
C2′—H2′*A*···O1	0.93	2.38	2.7116 (16)	101
C6′—H6′*A*···O2^{i}	0.93	2.43	3.2996 (18)	155

Symmetry code: (i) $-x+2, -y+1, -z+1$.

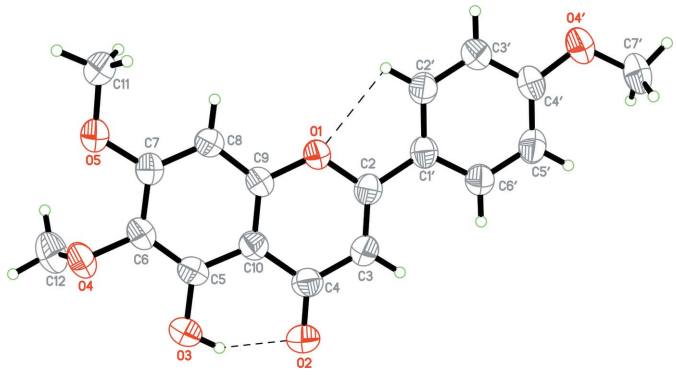


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate intramolecular hydrogen bonds.

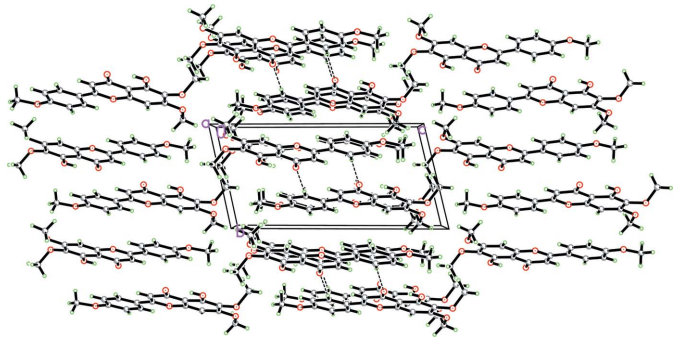


Figure 2
The crystal packing of (I), viewed down the *a* axis. Dashed lines indicate intermolecular hydrogen bonds.

H atoms were placed in calculated positions, with an O—H distance of 0.82 Å and C—H distances in the range 0.93–0.96 Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for the methyl and hydroxyl H atoms and $1.2U_{eq}$ for the remaining H atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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