

## Cirsimaritin

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## Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$

$R$  factor = 0.048

$wR$  factor = 0.157

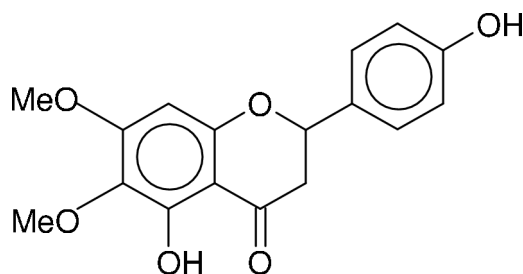
Data-to-parameter ratio = 18.8

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

The crystal structure of the title compound, 5,4'-dihydroxy-6,7-dimethoxyflavone,  $\text{C}_{17}\text{H}_{14}\text{O}_6$ , isolated from *Salvia nubicola*, is composed of chains that are stabilized by conventional ( $\text{O}-\text{H}\cdots\text{O}$  type) intramolecular and intermolecular hydrogen bonds with  $\text{O}\cdots\text{O}$  being 2.5780 (16) and 2.7459 (15)  $\text{\AA}$ , respectively. The benzopyran ring is essentially planar with hydroxyl and carbonyl O atoms lying close to its mean plane, the methoxyl groups inclined at 65.90 (11) and 8.39 (10) $^\circ$ , and the phenyl ring oriented at 23.30 (6) $^\circ$  from the benzopyran ring. The mean bond distances are:  $\text{O}-\text{C}_{\text{sp}^3}$  1.4372 (18),  $\text{O}-\text{C}_{\text{sp}^2}$  1.365 (12),  $\text{C}_{\text{sp}^2}-\text{C}_{\text{sp}^2}$  1.448 (17),  $\text{C}-\text{C}_{\text{aromatic}}$  1.393 (10) and  $\text{C}=\text{O}$  1.2559 (19)  $\text{\AA}$ .

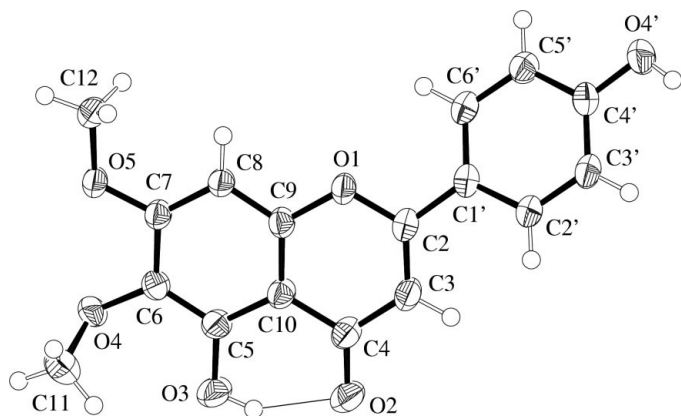
## Comment

*Salvia* is the largest genus of the plant family *Lamiaceae* (previously known as *Labiatae*) comprising aromatic and ornamental herbs and shrubs distributed in the temperate and tropical regions. Species of *Salvia* are known to contain terpenes and steroids (Pereda-Miranda *et al.*, 1986). We have isolated 5,4'-dihydroxy-6,7-dimethoxyflavone (cirsimaritin), (I), from *Salvia nubicola* Wall. *ex Sweet*; it had been previously isolated from *Centaurea scoparia* (Youssef & Frahm, 1995). The crystal structure of a very closely related flavone, eupatorin, has been reported from our laboratory (Parvez *et al.*, 2001). The inhibitory effects of (I) on both bovine-LAR (LAR = leukocyte antigen related) and rabbit platelet aggregation have been reported (Okada *et al.*, 1995). The crystal structure of (I) has been determined by X-ray crystallography and is reported in this paper.



(I)

The structure of (I) is presented in Fig. 1. The benzopyran ring is planar within 0.0314 (11)  $\text{\AA}$ . The mean plane of the phenyl ring  $\text{C}1'-\text{C}6'$  is inclined at 23.30 (6) $^\circ$  to the mean plane of the benzopyran ring. The carbonyl O2 and hydroxyl O3 lie slightly above [0.044 (2)  $\text{\AA}$ ] and below [0.066 (2)  $\text{\AA}$ ] the plane of the benzopyran ring. The methoxyl groups O4/C11 and O5/



**Figure 1**  
ORTEP (Johnson, 1976) drawing of (I), with displacement ellipsoids plotted at 50% probability level.

C12 are oriented at  $65.90(11)$  and  $8.39(10)^\circ$ , respectively, from the benzopyran ring. The molecular dimensions in (I) lie within expected values (Orpen *et al.*, 1994) for the corresponding bond distances and angles and agree well with those reported for a dozen or so similar compounds contained in the October 2001 release of the Cambridge Structural Database (Allen & Kennard, 1993). The mean bond distances are: O—C $_{sp^3}$  1.4372 (18), O—C $_{sp^2}$  1.365 (12), C $_{sp^2}$ —C $_{sp^2}$  1.448 (17), C—C $_{aromatic}$  1.393 (10) and C=O 1.2559 (19) Å.

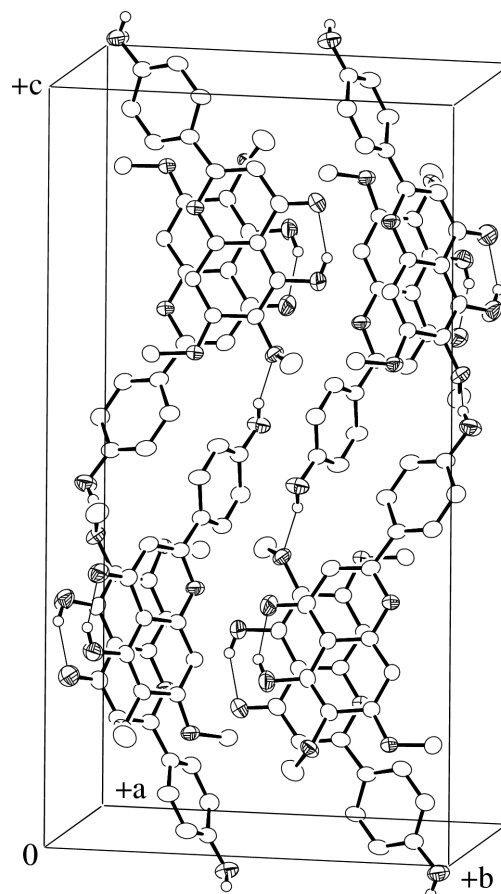
The structure is stabilized by strong intramolecular and intermolecular hydrogen bonds. A hydroxyl H atom is intramolecularly bonded with the carbonyl O atom [H3···O2 1.85, O3···O2 2.5780 (16) Å and O3—H3···O2 148°]. The intermolecular hydrogen bond between the 6-methoxyl O4 and the 4'-hydroxyl H atoms [H4'···O4 1.95, O4'···O4 2.7459 (15) Å and O4'—H4'···O4 164°] results in a chain structure along the *c* axis; details of the hydrogen-bonding geometry are provided in Table 2 and are presented in Fig. 2.

## Experimental

The plant material (all parts, 13 kg) was collected from Quetta, Pakistan, in July 1999, dried under shade for a period of two weeks, and soaked in hexane (12 l × 2) and methanol (12 l × 2) for 10 d in each solvent. Solvents were evaporated by vacuum distillation. The crude methanolic extract (217 g) was partitioned between water and ethyl acetate. The ethyl acetate layer was condensed (167 g) and subjected to column chromatography using hexane, hexane–ethyl acetate, ethyl acetate and ethyl acetate–methanol as mobile phases. Elution with 20% ethyl acetate in hexane afforded yellow crystals (11.6 mg) suitable for X-ray diffraction analysis.

### Crystal data

C <sub>17</sub> H <sub>14</sub> O <sub>6</sub>	Mo K $\alpha$ radiation
$M_r = 314.28$	Cell parameters from 3674 reflections
Orthorhombic, <i>Pbca</i>	$\theta = 1.0\text{--}30.0^\circ$
$a = 8.3669(2)$ Å	$\mu = 0.11\text{ mm}^{-1}$
$b = 13.5769(4)$ Å	$T = 293(2)$ K
$c = 25.2554(9)$ Å	Prism, yellow
$V = 2868.93(15)$ Å <sup>3</sup>	0.25 × 0.25 × 0.13 mm
$Z = 8$	
$D_x = 1.455\text{ Mg m}^{-3}$	



**Figure 2**  
Hydrogen-bonding pattern in (I), showing a hydrogen-bonded polymeric chain along the *c* axis.

### Data collection

Nonius KappaCCD diffractometer	2594 reflections with $I > 2\sigma(I)$
$\omega$ and $\varphi$ scans	$R_{int} = 0.036$
Absorption correction: multi-scan (SORTAV; Blessing, 1995, 1997)	$\theta_{max} = 30.0^\circ$
$T_{min} = 0.973$ , $T_{max} = 0.986$	$h = -11 \rightarrow 11$
6714 measured reflections	$k = -18 \rightarrow 18$
3958 independent reflections	$l = -35 \rightarrow 35$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.092P)^2 + 0.124P]$
$R[F^2 > 2\sigma(F^2)] = 0.048$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.157$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.98$	$\Delta\rho_{max} = 0.22\text{ e \AA}^{-3}$
3958 reflections	$\Delta\rho_{min} = -0.19\text{ e \AA}^{-3}$
211 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.004 (4)

**Table 1**

Selected geometric parameters (Å, °).

O1—C2	1.3645 (16)	O4—C11	1.4353 (19)
O1—C9	1.3794 (16)	O5—C7	1.3544 (16)
O2—C4	1.2559 (19)	O5—C12	1.4390 (17)
O3—C5	1.3502 (17)	O4'—C4'	1.3617 (17)
O4—C6	1.3821 (17)		
C2—O1—C9	119.77 (11)	C7—O5—C12	117.49 (12)
C6—O4—C11	115.94 (12)		

**Table 2**  
Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O4'-H4' \cdots O4^i$	0.82	1.95	2.7459 (15)	164
$O3-H3 \cdots O2$	0.82	1.85	2.5780 (16)	148

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

The H atoms were located from difference Fourier syntheses and were included in the refinement at geometrically idealized positions, with C—H 0.93–0.96 Å and O—H 0.82 Å, utilizing a riding model.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALE-PACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *TEXSAN* (Molecular Structure Corporation, 1994); software used to prepare material for publication: *SHELXL97*.

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