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Eupatorin

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The crystal structure of the title compound, 5,3′-dihydroxy-6,7,4′-trimethoxyflavone, $C_{18}H_{16}O_7$, isolated from *Ballota limbata* Linn., contains discrete molecules with normal molecular dimensions. The structure is stabilized by intramolecular and intermolecular hydrogen bonds between hydroxyl H atoms and carbonyl O atoms, with O···O interactions of 2.594 (2) and 2.673 (2) Å. The flavone moiety is essentially planar with methoxy methyl groups lying 0.169 (3)–0.851 (2) Å out of the plane.

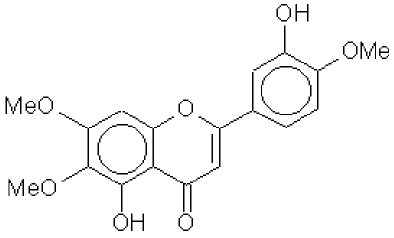
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.121
Data-to-parameter ratio = 11.7

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

Comment

Ballota limbata Linn. (Labiatae) is a small prickly shrub, found in the northern hilly areas of Pakistan. The chemical constituents of *B. limbata* have been investigated due to its uses in folk medicines (Watt, 1972). Eupatorin, (I), has been previously isolated from the cytotoxic extract of *Eupatorium semiserratum* DC. (Kupchan *et al.*, 1969). In this article, we describe the crystal structure of (I).



(I)

The crystal structure of (I) is composed of an essentially planar flavone moiety (Fig. 1), with a maximum deviation of 0.085 (1) Å for O1. The angles between the mean planes of the methoxy groups attached to C7, C4′ and C6, and the mean-plane of the flavone moiety are 7.01 (19), 2.9 (2) and 66.57 (12)°. The methyl C atoms C11, C12, and C13 lie at −0.851 (2), 0.219 (3) and 0.169 (3) Å from the mean plane of the flavone moiety. The bond distances and angles in the structure are normal and agree well with the corresponding values reported for ten similar compounds contained in the October 2000 updates of the Cambridge Structural Database (Allen & Kennard, 1993). The mean bond distances are: O—Csp^3 1.431 (3), O—Csp^2 1.359 (13), Csp^2—Csp^2 1.442 (18) and aromatic C—C 1.393 (12) Å, while C4═O2 is 1.266 (2) and C2═C3 is essentially a double bond with a distance of 1.352 (2) Å.

The crystal structure is stabilized by an intramolecular hydrogen bond involving a hydroxyl H atom and the carbonyl

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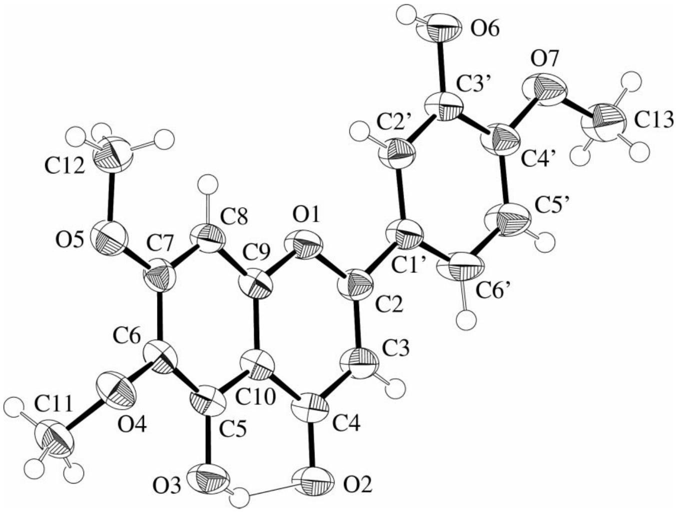

Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I) with 50% probability ellipsoids.

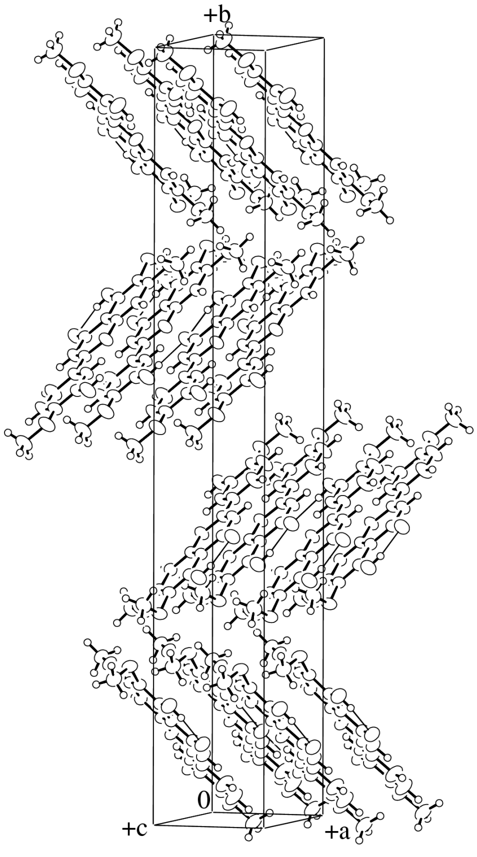

Figure 2
Unit-cell packing of (I).

O atom [O···O separation 2.594 (2) Å and O3—H3*A*···O2 angle of 148°] and an intermolecular hydrogen bond involving the other hydroxyl H atom and the carbonyl O atom of another molecule [O···O separation 2.673 (2) Å and O6—H6···O2^{i} angle of 174° [symmetry code: (i) $x - 1, y, z - 1$]. In the unit cell, the molecules stack parallel to each other in a herring-bone pattern as presented in Fig. 2.

Experimental

Ballota limbata plants were collected from the Mansehra district of North Western Frontier Province, Pakistan. The shade-dried and ground whole plant material (16 kg) was extracted with methanol. The resulting gummy material (1 kg) was partitioned into hexane, chloroform, ethyl acetate and *n*-butanol soluble fractions. The chloroform fraction was subjected to column chromatography using a hexane/chloroform and chloroform/methanol gradient system. The fraction using chloroform/methanol (9:1) over flash silica gel afforded (I) as yellow needles.

Crystal data

$C_{18}H_{16}O_7$
$M_r = 344.31$
Monoclinic, $P2_1/c$
$a = 5.5475$ (8) Å
$b = 37.690$ (5) Å
$c = 7.4742$ (6) Å
$\beta = 96.462$ (9)°
$V = 1552.8$ (3) Å^3
$Z = 4$
$D_x = 1.473$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 25$–50°
$\mu = 0.97$ mm^{-1}
$T = 293$ (2) K
Needle, yellow
0.40 × 0.20 × 0.07 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical ψ-scan (3 reflections; North *et al.*, 1968)
$T_{min} = 0.70$, $T_{max} = 0.94$
5869 measured reflections
2706 independent reflections
2310 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 68.0$°
$h = 0 \rightarrow 6$
$k = -45 \rightarrow 45$
$l = -8 \rightarrow 8$
3 standard reflections every 200 reflections intensity decay: 0.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.121$
$S = 1.09$
2706 reflections
232 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.068P)^2 + 0.387P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0092 (9)

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.359 (2)	C3′—O6	1.351 (2)
O1—C9	1.379 (2)	C4′—O7	1.354 (2)
C4—O2	1.266 (2)	O4—C11	1.427 (2)
C5—O3	1.348 (2)	O5—C12	1.431 (2)
C6—O4	1.374 (2)	O7—C13	1.435 (2)
C7—O5	1.351 (2)		
C2—O1—C9	119.97 (12)	C7—O5—C12	118.77 (13)
C6—O4—C11	114.93 (13)	C4′—O7—C13	117.25 (14)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O2	0.82	1.86	2.594 (2)	148
O6—H6···O2^{i}	0.82	1.86	2.673 (2)	174

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

The H atoms were located from difference maps and were placed at geometrically idealized positions (C—H 0.93 and 0.96 Å, and O—

H 0.82 Å) utilizing riding models, and torsional parameters were refined for each methyl group. The methyl and non-methyl H atoms were allowed isotropic displacement parameters 1.5 and 1.2 times, respectively, the displacement parameters of the atoms to which they were attached.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1994); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *TEXSAN*; software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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