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5,12-Dihydroxy-2,6,7,13-tetramethoxyflavone

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Key indicators

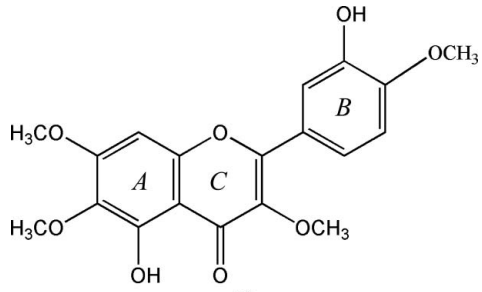
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.046
wR factor = 0.145
Data-to-parameter ratio = 12.3

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

The approximately planar title compound, $C_{19}H_{18}O_8$, containing a benzopyranone system, a benzene ring and four methoxy groups, is a natural flavone extracted from the herb *Vitex trifolia* L. Intermolecular O—H···O and possible C—H···O interactions result in the formation of a two-dimensional network.

Comment

The title compound, (I), is a natural flavone, which possesses analgesic and anti-inflammatory properties (Okuyama *et al.*, 1998). In addition, it inhibits the proliferation of lymphocytes and the growth of some cancer cells (You *et al.*, 1998; Kobayakawa *et al.*, 2004). In order to better correlate these biological effects with the detailed conformation of the molecule and its propensity to form specific intermolecular interactions, we report its structure here.



(I)

Compound (I) is built up from a benzopyranone system, a phenyl ring and four methoxy groups (Fig. 1). The atoms of the benzopyranone system, composed of rings *A* (C4–C9) and *C* (O1/C9/C1–C4), are almost coplanar, the dihedral angle between the rings being 2.7 (1)°. The benzene ring *B* (C10–C15) is rotated by 8.3 (1)° with respect to the benzopyranone framework. In the related molecule dimethylgenistein (Zhang *et al.*, 2005) the equivalent *B* ring is rotated by 56.28 (3)°, perhaps as a result of different neighboring hydrogen bonds.

The molecules of (I) are linked into an infinite two-dimensional network formed by a combination of O—H···O and possible C—H···O hydrogen bonds arising from terminal methyl groups (Fig. 2 and Table 1). There are also two intramolecular O—H···O bonds (Fig. 1 and Table 1.

Experimental

Compound (I) (16 mg) was isolated from the Chinese medical herb *Vitex trifolia* L. (500 mg) by high-speed counter-current chromatography with light petroleum–ethyl acetate–methanol–water (5:5:3:7,

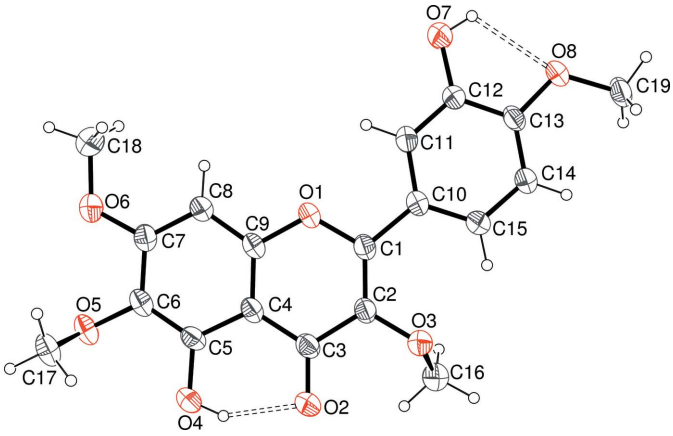


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids (arbitrary spheres for the H atoms). Hydrogen bonds are indicated by dashed lines.

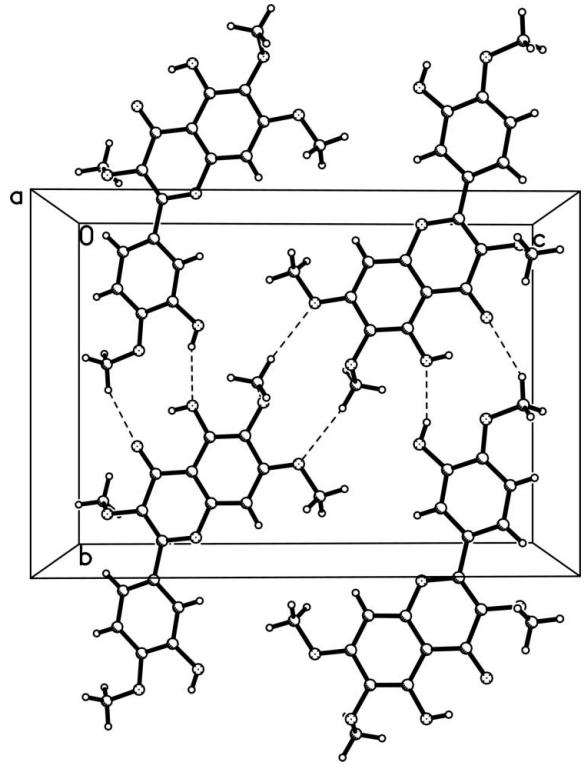


Figure 2
Part of the two-dimensional network in (I), with the O—H···O and C—H···O contacts shown as dashed lines.

v/*v*) as the two-phase solvent system. The product was recrystallized from dichloromethane at room temperature (yield 12 mg, 98.5%; m.p. 463 K). Analysis calculated for $C_{19}H_{18}O_8$: C 60.96, H 4.76%; found: C 60.94, H 4.75%. ^{1}H NMR (DMSO-*d*6, 400 MHz, p.p.m.): δ 12.59 (1 H, *s*, 5-OH), 9.46 (1 H, *s*, 12-OH), 6.85 (1 H, *s*, 8-H), 7.57 (1 H, *d*, 11-H), 7.08 (1 H, *d*, 14-H), 7.56 (1 H, *d*, 15-H), 3.71, 3.78, 3.85, 3.90 (12 H, OMe × 4). ^{13}C NMR (DMSO-*d*6, 100 MHz, p.p.m.): δ 157.23 (C-1), 143.42 (C-2), 183.72 (C-3), 111.03 (C-4), 157.11 (C-5), 137.02 (C-6), 164.13 (C-7), 96.76 (C-8), 161.07 (C-9), 125.86 (C-10), 120.49 (C-11), 151.80 (C-12), 155.76 (C-13), 117.26 (C-14), 127.64 (C-15), 65.49 (C-16), 65.16 (C-17), 61.92 (C-18), 61.08 (C-19).

Crystal data

$C_{19}H_{18}O_8$
M_r = 374.33
Monoclinic, $C2/c$
a = 15.528 (4) Å
b = 12.446 (3) Å
c = 17.672 (4) Å
β = 92.565 (4)°
V = 3411.7 (13) Å^3
Z = 8
D_x = 1.458 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
Block, colorless
0.24 × 0.21 × 0.19 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.973, T_{max} = 0.979
8808 measured reflections
3004 independent reflections
1569 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.145
S = 1.03
3004 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 1.0805P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7···O4^{i}	0.82	2.16	2.942 (3)	161
C17—H17*A*···O6^{ii}	0.96	2.53	3.482 (4)	171
C19—H19*A*···O2^{i}	0.96	2.51	3.334 (4)	144
O4—H4···O2	0.82	1.86	2.591 (3)	148
O7—H7···O8	0.82	2.24	2.681 (3)	115

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

H atoms were positioned geometrically (O—H = 0.82 Å and C—H = 0.93–0.96 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(methyl C,O).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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