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Key indicators

Single-crystal X-ray study
 $T = 297$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.040
 wR factor = 0.112
Data-to-parameter ratio = 16.2

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

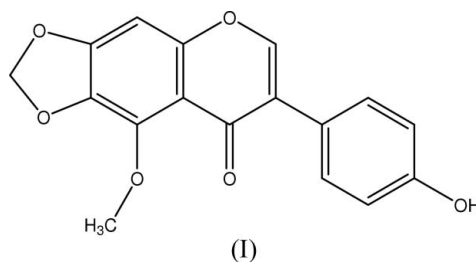
4'-Hydroxy-5-methoxy-6,7-methylenedioxyisoflavone

The title compound, $\text{C}_{17}\text{H}_{12}\text{O}_6$, forms an infinite one-dimensional zigzag-like chain developing parallel to the b axis through $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds. The chains are stacked along the c axis. The crystal structure is further stabilized by weak $\pi-\pi$ and $\text{C}-\text{H}\cdots\pi$ interactions.

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Comment

Iris germanica Linn. belongs to the family Iridaceae. *Iris* is a rhizomatous or bulbous herb. The genera include commercially valuable ornamentals. Historically, the plant was used to treat constipation, dermatitis and skin disease. *Iris* species are also reported to have various biological activities, including cytotoxic, anticancer, anti-ulcer, piscicidal and insecticidal activities (Miyake *et al.*, 1997; Takahashi *et al.*, 1999; Wong *et al.*, 1985, 1986; Muto *et al.*, 1994; Takahashi *et al.*, 1993). The plant contains isoflavones such as irisolidone, irisolidone 7- O - α - D -glucoside, irilone, iriflogenin, iriskashmirianin, irigenin and the title compound, nigricin, (I) (Wollenweber *et al.*, 2003; Orhan *et al.*, 2003; Atta-ur-Rahman *et al.*, 2003). The plant possesses anti-inflammatory and hypolipidemic activity (Atta-ur-Rahman *et al.*, 2003; Choudhary *et al.*, 2005). Nigricin has also been reported for its bactericidal activity against *Staphylococcus aureus* and *Pseudomonas aeruginosa* (Atta-ur-Rahman *et al.*, 2003).



A molecular view of (I) is shown in Fig. 1. Bond lengths and angles display normal values (Allen, 2002), comparable with related reported structures (Koh & Ng, 1993). The fused-ring system is almost planar, with a maximum deviation of 0.142 Å for atom C2. The hydroxyphenyl group (O6/C10–C15) attached at the C2 position is twisted away from the plane of the fused rings, with the dihedral angle between them being 49.80 (5)°. The methoxy group at the C4 position is almost coplanar with the plane of the C6–C8/C5/C4/C9 benzene ring, with a C17–O4–C4–C9 torsion angle of -170.75 (12)°. There are intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds in the crystal structure of (I), forming an infinite one-dimensional zigzag-like chain along the b axis (Table 1, Fig. 2). The chains are stacked along the c axis.

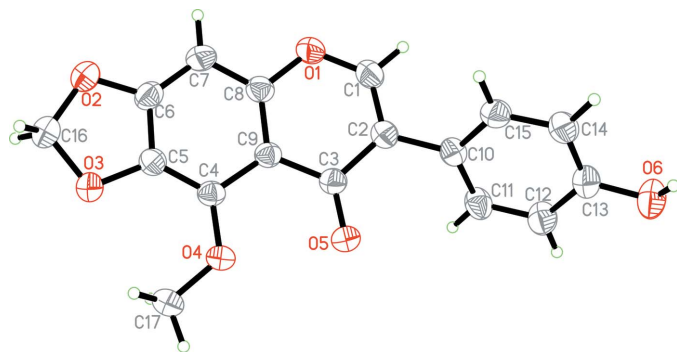


Figure 1
The molecular structure of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

The crystal structure is also stabilized by weak offset π - π interactions involving the C10–C15 benzene rings related by the symmetry operator $(1 - x, 2 - y, -z)$. The centroid–centroid separation is 3.8824 (9) Å with an interplanar distance of 3.699 (1) Å, and the angle between the ring normal and the centroid vector is 17.7°. The crystal structure is further stabilized by C–H $\cdots\pi$ interactions, involving the O1/C1–C3/C9/C8 ring (Table 1).

Experimental

The air-dried rhizomes of *Iris germanica* Linn. were crushed into a moderately coarse powder (3.0 kg) and this was defatted with hexane (12 l) at room temperature; the percentage yield of the plant was $4.76 \times 10^{-4}\%$. Plant material was extracted with ethanol–water (80:20, 25 \times 3 l) and the extract was vacuum-dried (765.00 g). The extract was further fractionated with 6.0 l dichloromethane (145 g), 6.0 l ethyl acetate (50 g) and 6.0 l *n*-butanol (202 g). The ethyl acetate extract (15.5 g) was subjected to silica-gel column chromatography with increasing polarities of petroleum ether–dichloromethane and then with methanol. The fraction obtained from pure dichloromethane (50.0 mg) was subjected to preparative thin-layer chromatography with a solvent system of 1% methanol–dichloromethane. The title compound, (I), was obtained in a yield of 14.3 mg ($4.76 \times 10^{-4}\%$, $R_F = 0.19$ in 1% methanol–dichloromethane) as yellowish crystals (m.p. 500–502 K).

Crystal data

$C_{17}H_{12}O_6$
 $M_r = 312.27$
Monoclinic, $P2_1/c$
 $a = 11.0058$ (7) Å
 $b = 10.1990$ (7) Å
 $c = 12.7693$ (8) Å
 $\beta = 106.922$ (1)°
 $V = 1371.27$ (15) Å³
 $Z = 4$

$D_x = 1.513$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 4562 reflections
 $\theta = 4.2$ – 28.3°
 $\mu = 0.12$ mm⁻¹
 $T = 297$ (2) K
Block, yellow
0.59 \times 0.54 \times 0.42 mm

Data collection

Bruker SMART APEX-2 CCD
area-detector diffractometer
 ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2005)
 $T_{min} = 0.866$, $T_{max} = 0.953$
10654 measured reflections

3396 independent reflections
2784 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.017$
 $\theta_{max} = 28.3^\circ$
 $h = -14 \rightarrow 14$
 $k = -13 \rightarrow 13$
 $l = -17 \rightarrow 17$

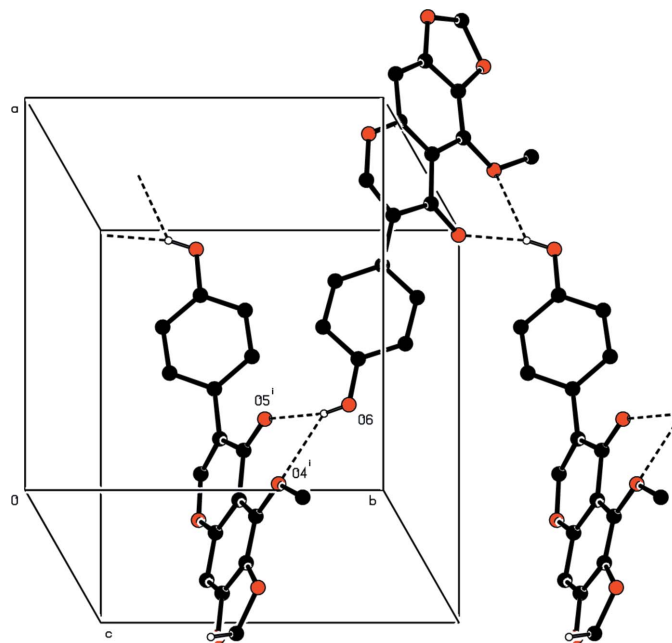


Figure 2
Part of the crystal packing of (I), showing the zigzag-like chain formed by O–H \cdots O interactions. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry code (i): $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$.]

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.112$
 $S = 1.05$
3396 reflections
209 parameters
H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.3602P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.25$ e Å⁻³
 $\Delta\rho_{min} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O6–H6A \cdots O4 ⁱ	0.82	2.49	3.0072 (15)	122
O6–H6A \cdots O5 ⁱ	0.82	1.96	2.7581 (17)	166
C17–H17C \cdots Cg ⁱⁱ	0.96	2.82	3.567	136

Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + 2, -y + 2, -z + 1$. Cg is the centroid of the O1/C1–C3/C9/C8 ring.

H atoms were placed in calculated positions, with an O–H distance of 0.82 Å and C–H distances in the range 0.93–0.97 Å. The $U_{iso}(H)$ values were constrained to be $1.5U_{eq}$ of the carrier atom for hydroxyl and methyl H atoms, and $1.2U_{eq}$ for the remaining H atoms.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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