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2-(5,7-Dihydroxy-4-oxo-4*H*-chromen-3-yl)-5-methoxy-1,4-benzoquinone (isoflavonequinone)

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In the title molecule, 5,7-dihydroxy-4′-methoxyisoflavonequinone, $C_{16}H_{10}O_7$, the dihedral angle between the chromene moiety and the cyclohexa-1,4-diene ring is 51.72 (5)°. The molecular structure is stabilized by an intramolecular O—H···O hydrogen bond. In the crystal structure, intermolecular O—H···O hydrogen bonds link the molecules to form chains along the *c* axis. Adjacent molecular chains are linked along the *b* axis by C—H···O hydrogen bonds to form a network.

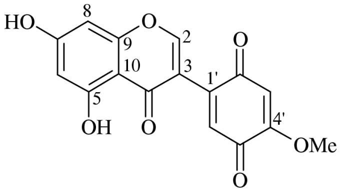
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.125
Data-to-parameter ratio = 15.2

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

Comment

The isoflavonoid compounds are common constituents of the plant family Leguminosae. These compounds exhibit anti-oxidant activity, *i.e.* an ability to reduce free radical formation and to scavenge free radicals (Pietta, 2000). The crystal structures of 4′,6,7-trimethoxyisoflavone, (II) (Shoja, 1992*a*), 7-hydroxy-4′-methoxyisoflavone, (III) (Shoja, 1992*b*), and 5,5′-dihydroxy-7,3′,4′-trimethoxyisoflavone, (IV) (Ngounou *et al.*, 2000), have been reported. In the present study, the structure of 5,7-dihydroxy-4′-methoxyisoflavonequinone [systematic name: 2-(5,7-dihydroxy-4-oxo-4*H*-chromen-3-yl)-5-methoxy-1,4-benzoquinone], (I), has been investigated.



(I)

The molecular structure of (I), with the atom-numbering scheme, is shown in Fig. 1. In the chromene ring system, the pyran ring is slightly distorted from coplanarity with the benzene ring, and the dihedral angle formed by these rings is 3.33 (6)°. The methoxyquinone substituent is twisted from the chromene ring system, with the C4—C3—C1′—C6′ torsion angle being 54.4 (2)°; the dihedral angle between the least-squares planes through the chromene moiety and the cyclohexa-1,4-diene ring is 51.72 (5)°. The corresponding dihedral angles for the other above-mentioned isoflavone structures are −42.8 (5)° for (II) (Shoja, 1992*a*), 46.6 (11)° for (III) (Shoja, 1992*b*), and −37.4 (8) and 38.2 (8)° for the two independent molecules of (IV) (Ngounou *et al.*, 2000). The methoxy group at C4′ lies in the plane of the quinone ring, the C5′—C4′—O6—C11 torsion angle being −2.0 (2)°.

As observed in other flavone structures, the hydroxyl group at C5 is involved in an intramolecular O—H···O hydrogen

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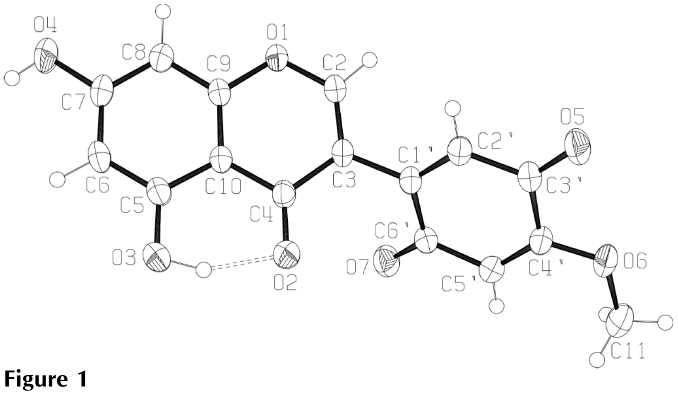

Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids. The intramolecular hydrogen bond is indicated by an open dashed line.

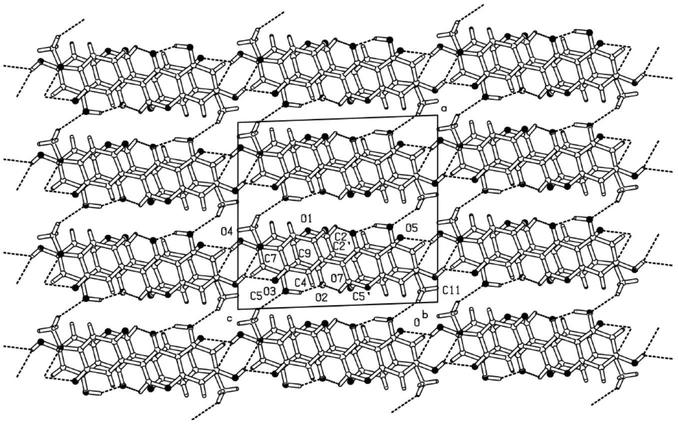

Figure 2
Packing of the molecules of the title compound, viewed down the *b* axis.

bond with carbonyl atom O2 at C4 (Table 2). In the crystal, the molecules translated by one unit along the *c* axis are linked by O4—H4O···O5^{i} and O4—H4O···O6^{i} [symmetry code: (i) *x*, *y*, *z* + 1] hydrogen bonds to form molecular chains. Along the *b* axis, screw-related molecules in adjacent chains are linked by C2—H2···O2^{ii} [symmetry code: (ii) $\frac{1}{2} - x$, $\frac{1}{2} + y$, $1 - z$] hydrogen bonds to form a molecular network (Fig. 2). Adjacent layers are interlinked through weak C11—H11*A*···O3^{iii} [symmetry code: (iii) $-x$, $-y$, $1 - z$] interactions. The methyl atom C11 is involved in a short intermolecular contact [2.890 (2) Å] with atom O4^{iv} [symmetry code: (iv) $\frac{1}{2} + x$, $\frac{1}{2} - y$, $-1 + z$].

Experimental

The title compound was isolated from the heartwood of *Dalbergia cochinchinensis*. Crystals (decomposing at 523 K) were obtained by recrystallization from acetone of the crude extract obtained from ethyl acetate. Spectroscopic analysis, ^{1}H NMR (DMSO-d_6, δ): 3.90 (*s*, 3H, OCH$_3$), 6.15 (*s*, 1H, H-3′), 6.31 (*d*, 1H, *J* = 2.13 Hz, H-8), 6.45 (*d*, 1H, *J* = 2.13 Hz, H-6), 7.14 (*s*, 1H, H-6′), 8.30 (*s*, 1H, H-2), 12.6 (*s*, 1H, OH at C-5); ^{13}C NMR (DMSO-d_6), δ): 56.5 (OCH$_3$), 94.1 (C-8), 99.4 (C-6), 103.9 (C-10), 107.9 (C-3′), 115.4 (C-3), 133.2 (C-6′), 137.9 (C-1′), 157.1 (C-4′), 157.4 (C-2), 158.4 (C-9), 161.1 (C-5), 164.7 (C-7), 178.7 (C-4), 181.4 (C-5′), 184.9 (C-2′). LREIMS *m*/*z* 314 (calculated for $C_{16}H_{10}O_7$).

Crystal data

$C_{16}H_{10}O_7$
$M_r = 314.24$
Monoclinic, $P2_1/a$
$a = 12.6447\ (2)$ Å
$b = 7.7044\ (1)$ Å
$c = 13.5573\ (1)$ Å
$\beta = 92.103\ (1)°$
$V = 1319.86\ (3)$ Å^3
$Z = 4$
$D_x = 1.581$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 75 reflections
$\theta = 1.5–30.5°$
$\mu = 0.13$ mm^{-1}
$T = 295\ (2)$ K
Needle, yellow
0.5 × 0.2 × 0.1 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.970$, $T_{max} = 0.987$
9429 measured reflections
3781 independent reflections
2899 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 30.5°$
$h = -17 \rightarrow 15$
$k = -10 \rightarrow 10$
$l = -16 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.125$
$S = 1.02$
3781 reflections
248 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 0.2705P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Table 1
Selected interatomic distances (Å).

O1—C2	1.3513 (14)	O6—C4′	1.3453 (14)
O1—C9	1.3761 (14)	O6—C11	1.4513 (19)
O2—C4	1.2448 (15)	O7—C6′	1.2223 (15)
O3—C5	1.3442 (16)	C2—C3	1.3428 (17)
O4—C7	1.3559 (16)	C1′—C2′	1.3442 (17)
O5—C3′	1.2221 (16)	C4′—C5′	1.3364 (18)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3O···O2	0.95 (3)	1.69 (3)	2.582 (1)	155 (2)
O4—H4O···O5^{i}	0.84 (3)	2.24 (3)	2.879 (2)	133 (2)
O4—H4O···O6^{i}	0.84 (3)	2.33 (3)	3.071 (2)	147 (2)
C2—H2···O2^{ii}	0.93 (2)	2.47 (2)	3.006 (2)	117 (1)
C11—H11*A*···O3^{iii}	1.00 (2)	2.60 (2)	3.510 (2)	151 (2)
C11···O4^{iv}			2.890 (2)	

Symmetry codes: (i) $x, y, 1 + z$; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, 1 - z$; (iii) $-x, -y, 1 - z$; (iv) $\frac{1}{2} + x, \frac{1}{2} - y, -1 + z$.

H atoms were located in a difference Fourier map and both positional and isotropic displacement parameters were refined. The ranges of C—H and O—H bond lengths are 0.93 (2)–1.02 (2) and 0.84 (3)–0.95 (3) Å, respectively. Owing to a large fraction of very weak data at higher angles, the completeness of the data is low (94.27%).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SHELXTL* (Siemens, 1996) and *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990) and *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *PLATON* and *SHELXL*97.

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