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8-Hydroxy-3,4-dimethylisocoumarin

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Key indicators

Single-crystal X-ray study
T = 81 K
Mean σ(C–C) = 0.004 Å
R factor = 0.060
wR factor = 0.128
Data-to-parameter ratio = 12.4

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

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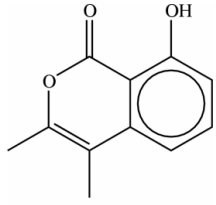
The title compound, $C_{11}H_{10}O_2$, known as oospolactone, is a simple isocoumarin which has been isolated as a secondary metabolite from a culture extract of the fungi *Gloeophyllum subferrugineum* and *G. sepiarium*. The molecular core is planar, with an intramolecular hydrogen bond between the hydroxyl and ketone groups.

Comment

Isocoumarins are an interesting class of compounds which are very important in biological systems. Naturally occurring oospolactone has been a molecule of interest since its isolation (Yamamoto, 1961) and identification (Yamamoto *et al.*, 1961). It has been widely studied and synthesized as an antifungal and anticancer agent (e.g. Kim *et al.*, 2002; Agata *et al.*, 2000; Sonnenbichler *et al.*, 1993; Singh & Singh, 1988; Nozawa *et al.*, 1981; Chatterjea *et al.*, 1980; Nakajima *et al.*, 1976).



(I)

In our studies, the isocoumarin oospolactone was isolated from a culture of *G. subferrugineum* and *G. sepiarium*; the molecule is shown in Fig. 1. A search of the Cambridge Structural Database (CSD; Allen, 2002; Version 5.24, November 2002) shows only 18 related isocoumarins. The molecular cores in these isocoumarins are all similar and 5,6,8-trimethoxy-3,4,7-trimethylisocoumarin (Botha *et al.*, 1991)

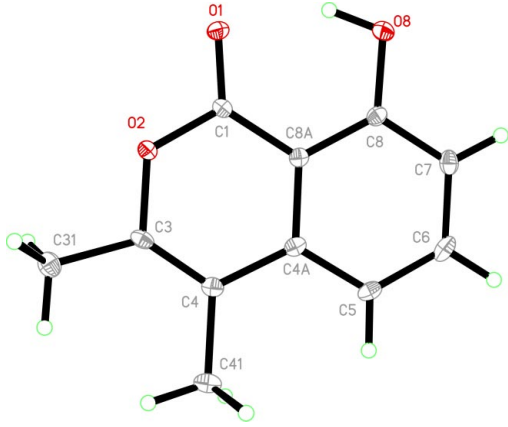


Figure 1
The molecular structure of oospolactone. Atomic displacement ellipsoids are drawn at the 30% probability level.

resembles oospolactone most closely. The bond lengths and angles in this synthetic compound are similar to those in oospolactone [O1—C1 = 1.204 (3) and 1.226 (3) Å; C3—C4 = 1.330 (3) and 1.336 (4) Å; O1—C1—O2 = 115.1 (2) and 116.6 (3)°]. The most notable feature of oospolactone is the intramolecular hydrogen bonding between the hydroxyl group and the lactone (see Table 1). This can be seen in other isocoumarins possessing an 8-hydroxy group with respect to the lactone [*e.g.* CSD refcode FLOCOS, oospolactone (1.725 Å; Brisse *et al.*, 1978), BIYVAG (1.817 Å; Schmalle *et al.*, 1982) and RAFDEH (1.917 Å; Krohn *et al.*, 1997)]. However, with the exception of FLOCOS, this oospolactone shows the closest intramolecular interaction of all the 8-hydroxyisocoumarins.

Experimental

Oospolactone was purified from the culture extracts of *G. subferrugineum* and *G. sepiarium*. The extracts were dissolved in acetonitrile and oospolactone was precipitated by the addition of an equal amount of water. The precipitate was further purified using an Agilent 1090 HPLC equipped with a 150 × 4.6 mm phenyl–hexyl column (Phenomenex, Torrance, CA) and UV–Vis diode array detector. Elution was performed using a linear gradient of 30% acetonitrile in water to 100% acetonitrile with a flow rate of 1.0 ml min^{-1}. The compound was crystallized from acetonitrile at room temperature.

Crystal data

$C_{11}H_{10}O_3$
M_r = 190.19
Monoclinic, $C2/c$
a = 11.283 (2) Å
b = 15.457 (3) Å
c = 10.365 (2) Å
β = 102.56 (3)°
V = 1764.3 (6) Å^3
Z = 8
D_x = 1.432 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 356 reflections
θ = 2.3–19.1°
μ = 0.10 mm^{-1}
T = 81 (2) K
Plate, colorless
0.15 × 0.10 × 0.06 mm

Data collection

Bruker–Siemens SMART APEX diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.985, T_{max} = 0.994
10791 measured reflections
1650 independent reflections
1057 reflections with $I > 2\sigma(I)$
R_{int} = 0.116
θ_{max} = 25.5°
$h = -13 \rightarrow 13$
$k = -18 \rightarrow 18$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.128
S = 1.03
1650 reflections
133 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0349P)^2 + 1.8671P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O8—H8···O1	0.92 (4)	1.80 (4)	2.628 (3)	148 (3)

Atom H8 was located in a difference Fourier map and freely refined. All other H atoms were positioned geometrically and refined using a riding model, with U_{iso} values constrained to be $1.2U_{eq}$ of the carrier atom and C—H = 0.95–0.98 Å.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *XS* in *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *XL* in *SHELXTL*; molecular graphics: *XP* in *SHELXTL*; software used to prepare material for publication: *XCIF* in *SHELXTL*.

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