Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-(2,3-Dihydroxy-3-methylbutoxy)-7*H*-furo[3,2-*g*]chromen-7-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.043
wR factor = 0.113
Data-to-parameter ratio = 12.1

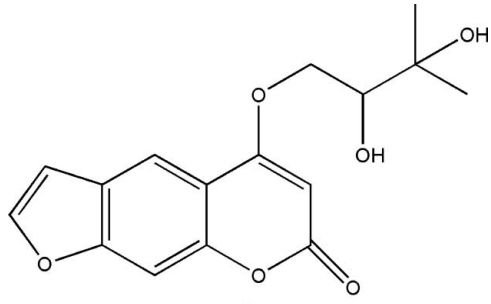
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 12 December 2006
Accepted 16 January 2007

In the title compound, $C_{16}H_{16}O_6$, the furan, benzene and pyrone rings are almost coplanar. The crystal structure is stabilized by intermolecular O—H···O and C—H···O hydrogen bonding.

Comment

The title compound (common name oxypeucedanin hydrate), (I), was isolated from a Chinese medicine, Radix Angelicae dahuricae (the root of *Angelica Dahurica*). Pharmacology experiments have shown that oxypeucedanin hydrate inhibits histamine release (Kimura *et al.*, 1997). We report here the crystal structure of (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The furan, benzene and pyrone rings are essentially coplanar, with a maximum deviation of 0.0174 (3) Å for atom C1. The dihedral angle between the coumarin unit and the furan ring is 1.1 (2)°. Adjacent molecules are linked *via* classical O—H···O hydrogen bonding and weak C—H···O hydrogen bonding (Table 1).

Experimental

The dried root of *Angelica Dahurica* was extracted with ethanol and fractionated into EtOAc- and water-soluble fractions. The EtOAc-soluble fraction was subjected to silica gel column chromatography using a $CHCl_3$–MeOH gradient (1:0 to 0:1) to obtain oxypeucedanin hydrate, which was further purified by preparative thin-layer chromatography. Single crystals of (I) were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{16}H_{16}O_6$
M_r = 304.29
Monoclinic, $P2_1/c$
a = 9.1301 (19) Å
b = 10.015 (2) Å
c = 16.003 (3) Å
β = 98.824 (4)°
V = 1445.9 (5) Å^3
Z = 4
D_x = 1.398 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (2) K
Block, colourless
0.24 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
7192 measured reflections
2549 independent reflections
1511 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.113$
$S = 1.01$
2549 reflections
210 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.2072P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5A—H5A···O6^{i}	0.85	1.98	2.822 (3)	171
O6—H6A···O3^{ii}	0.87	1.97	2.819 (3)	167
C10—H10···O3^{iii}	0.93	2.58	3.479 (3)	162

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y+1, -z+2$; (iii) $-x+2, y+\frac{1}{2}, -z+\frac{5}{2}$.

The O5 hydroxy group is disordered over two positions; occupancies were refined and converged to 0.594 (3) and 0.406 (3), and were fixed at 0.6 and 0.4 in the final cycles of refinement. H atoms on O atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, with $U_{iso}(H) = 1.5U_{eq}(O)$. Methyl H atoms were placed in calculated positions with C—H = 0.96 Å and torsion angles were refined to fit the electron density, $U_{iso}(H) = 1.5U_{eq}(C)$. Other H atoms were placed in calculated positions, with C—H = 0.93 (aromatic), 0.97 (methylene) or 0.98 Å (methine), and refined in riding mode, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*.

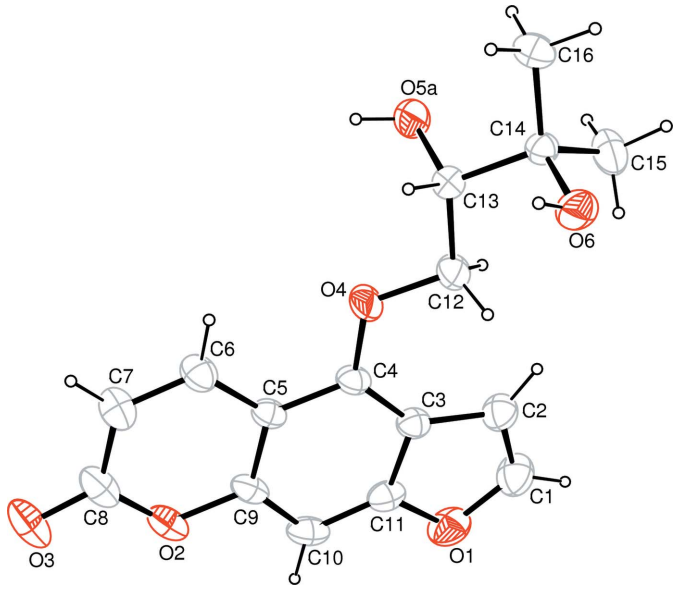


Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). The minor disordered component has been omitted for clarity.

References

Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kimura, Y., Okuda, H. & Baba, K. (1997). *J. Nat. Prod.* **60**, 249–251.
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