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1,3,8-Trihydroxy-6-methylanthraquinone monohydrate

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Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.005 Å
R factor = 0.080
wR factor = 0.168
Data-to-parameter ratio = 11.1

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

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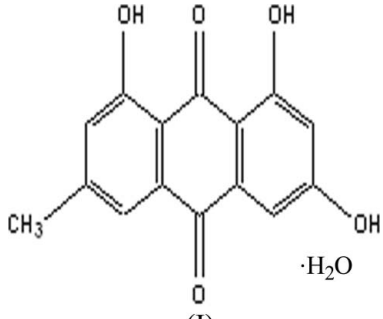
The title compound, $C_{15}H_{10}O_5\cdot H_2O$, also known as emodin monohydrate, was isolated from the roots of *Polygonum cuspidatum* Sieb. et Zucc. It is an anthraquinone derivative, and all of its non-H atoms are essentially coplanar. Intramolecular O—H···O hydrogen bonds and intermolecular O—H···O and C—H···O hydrogen bonds are observed in this crystal form. The crystal structure is further stabilized by weak π–π interactions and hydrogen bonds to the water molecule.

Comment

Pigments extracted from the roots of *Polygonum cuspidatum* (most common familiar name: Japanese knotweed) are closely related to an ancient class of natural anthraquinone-based colorants known as madder colours. In modern medicine they have been used to treat bacterial and viral infections, coughs, asthma, hypertension, and cancer (Chang *et al.*, 2005). One pigment component found in *Polygonum cuspidatum* Sieb. et Zucc., 1,3,8-trihydroxy-6-methylanthraquinone, is known as emodin and exhibits a variety of potent biological effects such as inhibition of growth of the murine leukemia virus (Kawai *et al.*, 1984), antimicrobial activity (Brown, 1980), an immunosuppressive effect (Huang *et al.*, 1992) and vasorelaxant activity (Huang, Chu & Chao, 1991; Huang, Lee *et al.*, 1991). We report here the structure of the monohydrate, (I),



(I)

The C—C bond lengths in (I) show normal values (Allen *et al.*, 1987), and the C—O and C═O bond lengths are comparable to those observed in similar structures (Ng *et al.*, 2005; Boonnak *et al.*, 2005). The non-H atoms of the organic molecule are essentially coplanar (Fig. 1), with an r.m.s. deviation of 0.013 Å. All O atoms are involved in hydrogen bonding (Table 1). In addition, the crystal structure is further stabilized by weak π–π interactions between the anthraquinone ring systems of the stacked molecules which lie approximately parallel to the (102) crystal planes (Fig. 2) and by two hydrogen bonds formed with the water molecule.

Experimental

The dried roots of *Polygonum cuspidatum* were ground to a powder. A sample of the powder (100 g) was extracted with 95% ethanol (500 ml) for 5 h with stirring. This extraction procedure was repeated three times. The extracts were combined and evaporated to dryness under reduced pressure, which yielded 11.3 g of crude extracts. The crude extracts were separated with light petroleum–ethyl acetate–methanol–water (3:5:4:6 *v*/*v*) using high-speed counter-current chromatography (HSCCC) to obtain 1,3,8-trihydroxy-6-methylanthraquinone (yield 35.3 mg; m.p. 529–530 K). Single crystals of the monohydrate were grown from undried acetone.

Crystal data

$C_{15}H_{10}O_5\cdot H_2O$
$M_r = 288.25$
Monoclinic, $P2_1/c$
$a = 9.570\ (3)$ Å
$b = 15.142\ (3)$ Å
$c = 9.245\ (3)$ Å
$\beta = 113.140\ (6)^\circ$
$V = 1231.9\ (6)$ Å^3
$Z = 4$
$D_x = 1.554$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.12$ mm^{-1}
$T = 153\ (2)$ K
Block, orange
0.30 × 0.14 × 0.14 mm

Data collection

Rigaku Mercury diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{min} = 0.728$, $T_{max} = 0.983$
11871 measured reflections
2256 independent reflections
1732 reflections with $I > 2\sigma(I)$
$R_{int} = 0.064$
$\theta_{max} = 25.3^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.080$
$wR(F^2) = 0.168$
$S = 1.17$
2256 reflections
203 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 1.359P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1	0.84	1.83	2.568 (3)	146
O3—H3···O5^{i}	0.84	2.55	3.090 (3)	123
O4—H4···O6^{ii}	0.84	1.79	2.620 (4)	172
O5—H5···O1	0.84	1.85	2.583 (3)	145
O5—H5···O1^{i}	0.84	2.52	3.021 (3)	119
O6—H6*A*···O2	0.86 (2)	1.99 (3)	2.804 (4)	158 (5)
O6—H6*B*···O3^{iii}	0.85 (2)	2.11 (4)	2.879 (4)	149 (7)
C12—H12···O4^{iv}	0.95	2.51	3.434 (4)	163

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

H atoms bound to C atoms were positioned geometrically and included in the refinement in the riding-model approximation [C—H = 0.95, 0.98, 0.99 and 1.00 Å for aromatic, methyl and aliphatic CH_2 and CH groups, respectively, and $U_{iso}(H) = 1.5U_{eq}$(methyl C) and $1.2U_{eq}(C)$].

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2000) and *PLATON* (Spek, 2003).

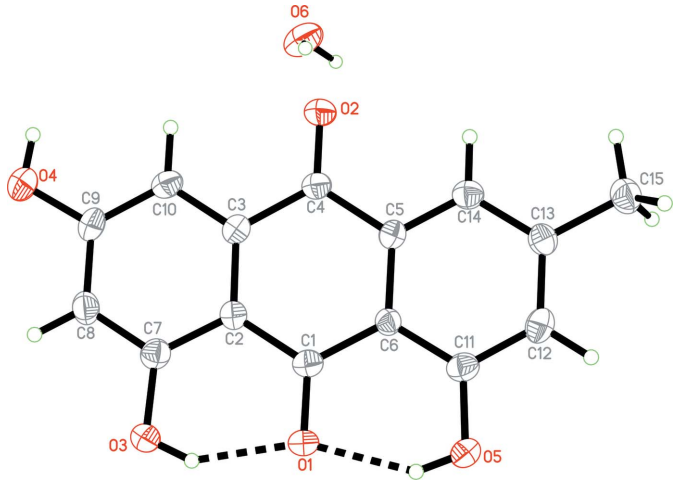


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-labelling scheme. H atoms are represented by small spheres of arbitrary radius. Dashed lines indicate hydrogen bonds.

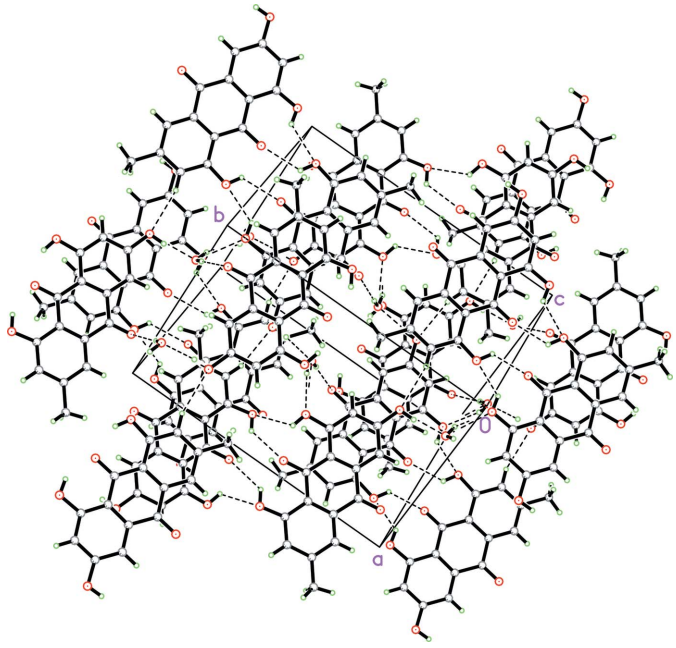


Figure 2
Packing diagram of (I), viewed down an axis perpendicular to the plane of the anthraquinone system of one molecule, which is essentially parallel to the (102) crystal plane. Hydrogen bonds are shown as dashed lines.

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