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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.045
 wR factor = 0.148
 Data-to-parameter ratio = 16.0

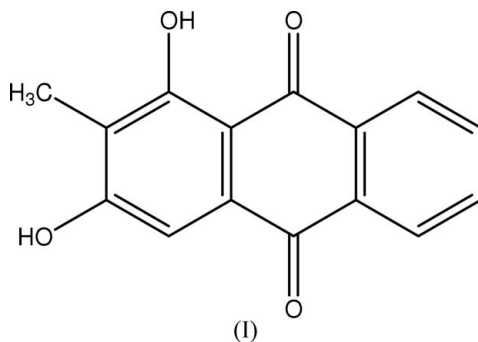
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

1,3-Dihydroxy-2-methyl-9,10-anthraquinone

The non-H atoms of the title compound, $\text{C}_{15}\text{H}_{10}\text{O}_4$, which was isolated from the roots of *Prismatomeris malayana* Ridl., are coplanar. Intramolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds are observed in the molecular structure. The molecules form centrosymmetric hydrogen-bonded dimers *via* intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds. The crystal structure is further stabilized by weak $\pi-\pi$ interactions.

Comment

Prismatomeris malayana Ridl ('Kradook Kai' in Thai) is a medicinal plant. The extract from the root of this plant has been used as a folk medicine for the treatment of skin diseases (Perry, 1980). The title compound, rubiadin, (I), has been isolated from the roots of *Prismatomeris malayana* Ridl. which were collected from Phuket province in the southern part of Thailand. Rubiadin was previously isolated from *Rubia cordifolia* (Tripathi *et al.*, 1997) and *Hedyotis capitellata* (Ahmad *et al.*, 2005). It possesses an anti-oxidant property which is better than that of EDTA, Tris, mannitol, vitamin E and *p*-benzoquinone (Tripathi *et al.*, 1997). As part of our systematic studies on chemical constituents of Thai medicinal plants (Chantrapromma *et al.*, 2003, 2004, 2005; Boonnak *et al.*, 2005; Fun *et al.*, 2005; Ng *et al.*, 2005), we have undertaken the X-ray crystal structure analysis of (I) in order to establish its molecular structure and relative stereochemistry.



The C—C bond lengths in (I) show normal values (Allen *et al.*, 1987). The C—O and C=O bond lengths (Table 1) are comparable to those observed in similar structures (Cotterill *et al.*, 1995; Ohsawa & Ohba, 1993). The non-H atoms of (I) are coplanar (Fig. 1), with a maximum deviation of 0.103 (1) Å for atom O4.

All O atoms are involved in hydrogen bonding. Atoms O1, O2 and O3 are involved in intramolecular $\text{O2}-\text{H2A}\cdots\text{O1}$ and $\text{C15}-\text{H15A}\cdots\text{O3}$ hydrogen bonds (Table 2), respectively, while atom O3 forms an intermolecular hydrogen bond with atom O4, *viz.* $\text{O3}-\text{H3A}\cdots\text{O4}^i$ [symmetry code: (i) $-x, 1-y,$

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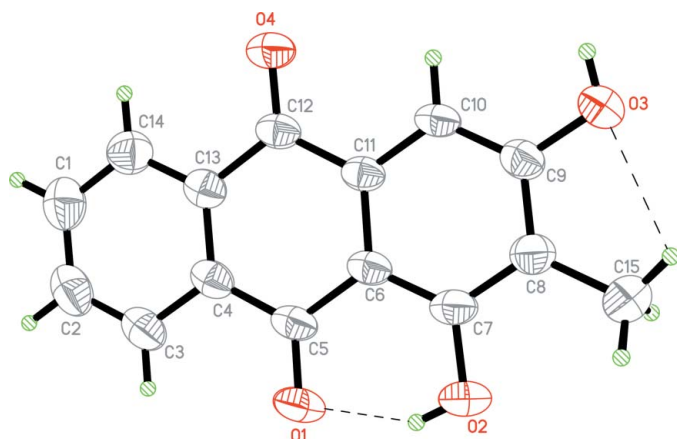


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Hydrogen bonds are shown as dashed lines.

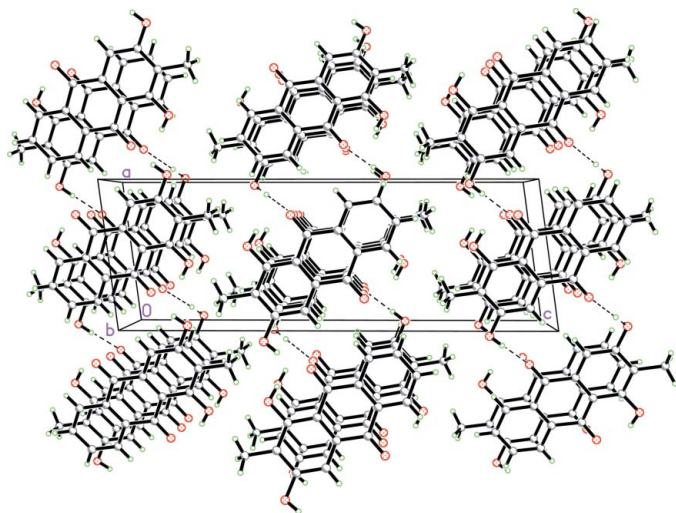


Figure 2
The crystal packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

1 - *z*], leading to the formation of a dimer (Table 2). In addition, the crystal packing is stabilized by weak π - π interactions between the anthraquinone ring system of inversion-related molecules stacked along the *b* axis (Fig. 2). The ring system is stacked in such a way that the centroid-centroid distance between the C1-C4/C13/C14 ring at (*x*, *y*, *z*) and the C6-C11 ring at (1 - *x*, 1 - *y*, 1 - *z*) is 3.640 (1) Å, while the centroid-centroid separation between the C4-C6/C11-C13 rings at (*x*, *y*, *z*) and (1 - *x*, 1 - *y*, 1 - *z*) is 3.593 (1) Å.

Experimental

Air-dried roots of *P.malayana* were ground and extracted with CH_2Cl_2 at room temperature. The residue obtained after evaporation of the solvent was repeatedly subjected to column chromatography over silica gel to afford (I). Single crystals of (I) were obtained by recrystallization from a CHCl_3 - CH_3OH (4:1 *v/v*) solvent system (m.p. 576-578 K).

Crystal data

$\text{C}_{15}\text{H}_{10}\text{O}_4$
 $M_r = 254.23$
 Monoclinic, $P2_1/c$
 $a = 7.3517$ (1) Å
 $b = 7.3268$ (1) Å
 $c = 21.1186$ (4) Å
 $\beta = 97.610$ (1)°
 $V = 1127.52$ (3) Å³
 $Z = 4$

$D_x = 1.498$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 4302 reflections
 $\theta = 2.0$ - 28.3 °
 $\mu = 0.11$ mm⁻¹
 $T = 293$ (2) K
 Plate, light brown
 $0.78 \times 0.41 \times 0.08$ mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.828$, $T_{\max} = 0.992$
 11402 measured reflections

2798 independent reflections
 2066 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 $\theta_{\max} = 28.3$ °
 $h = -9 \rightarrow 9$
 $k = -9 \rightarrow 9$
 $l = -26 \rightarrow 28$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.148$
 $S = 1.06$
 2798 reflections
 175 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0833P)^2 + 0.1198P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.24$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O1-C5	1.2408 (14)	O3-C9	1.3566 (15)
O2-C7	1.3501 (14)	O4-C12	1.2255 (14)
O2-C7-C6	121.55 (11)	O3-C9-C8	116.73 (12)
O2-C7-C8	116.54 (12)	O3-C9-C10	121.46 (11)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> -H... <i>A</i>	<i>D</i> -H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> -H... <i>A</i>
O2-H2A...O1	0.82	1.84	2.567 (2)	147
C15-H15A...O3	0.96	2.41	2.784 (2)	103
O3-H3A...O4 ⁱ	0.82	1.98	2.796 (1)	171

Symmetry code: (i) -*x*, -*y* + 1, -*z* + 1.

H atoms were placed in calculated positions, with O-H = 0.82 Å and C-H = 0.93 or 0.96 Å. The U_{iso} values were constrained to be $1.5U_{\text{eq}}$ of the carrier atom for hydroxyl and methyl H atoms and $1.2U_{\text{eq}}$ 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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