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3-(3,7-Dimethylocta-2,6-dienyloxy)-1,8-dihydroxy-6-methyl-9,10-anthraquinone

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Key indicators

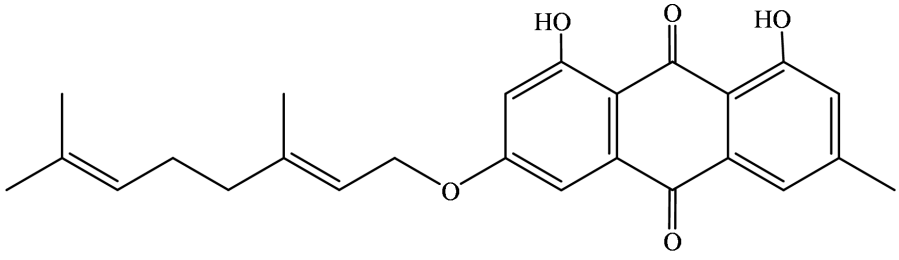
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.086
wR factor = 0.219
Data-to-parameter ratio = 13.4

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

The 9,10-anthraquinone ring system of the title compound, $C_{25}H_{26}O_5$, is essentially planar. The *O*-geranyl substituent is at the *meta* position with respect to the hydroxyl group. O—H···O intramolecular hydrogen bonds are observed in the molecular structure. The molecules form centrosymmetric hydrogen-bonded dimers *via* intermolecular C—H···O hydrogen bonds which generate rings of motif $R_2^2(10)$. The crystal structure is further stabilized by weak π–π interactions.

Comment

Cratoxylum is a small genus belonging to the *Guttiferae* family and found mainly in Southeast Asia (Robson, 1974); it has been used in traditional medicine (Usher, 1984) and some species of this genus exhibit antimalarial and antiprotozoal properties and also are slightly cytotoxic against human L6 cells (Seo *et al.*, 2002; Zakaria, 2004). In the course of our studies involving bioactive compounds from medicinal plants, the structure determination of the title compound, (I), was undertaken. We have isolated (I) for the first time from *Cratoxylum formosum ssp. pruniflorum*, widely distributed in the northeastern part of Thailand. It was previously isolated from *Psorospermum febrifugum* (Botta *et al.*, 1983).



(I)

The 9,10-anthraquinone ring system in (I) is essentially planar (Fig. 1), with a maximum deviation of 0.068 (3) Å for atom C10. The *O*-geranyl substituent (O5/C15–C23) is at the *meta* position with respect to the C14 hydroxyl group and the C15—O5—C12—C11 torsion angle of 178.7 (3)° indicates a (+)-antiperiplanar conformation.

The bond lengths in (I) show normal values (Allen *et al.*, 1987). Intramolecular O—H···O and intermolecular C—H···O interactions are observed (Table 2). The molecules are linked together to form dimers by C4—H4*A*···O3^{i} interactions [symmetry code: (i) $-x$, $-y$, $-z$] (Fig. 2). In the molecular structure, both O1—H1···O2 and O4—H4···O2 intramolecular hydrogen bonds involving the hydroxyl groups generate $R_2^1(6)$ ring motifs (Etter *et al.*, 1990). The molecules, which are linked together to form dimers by C—H···O

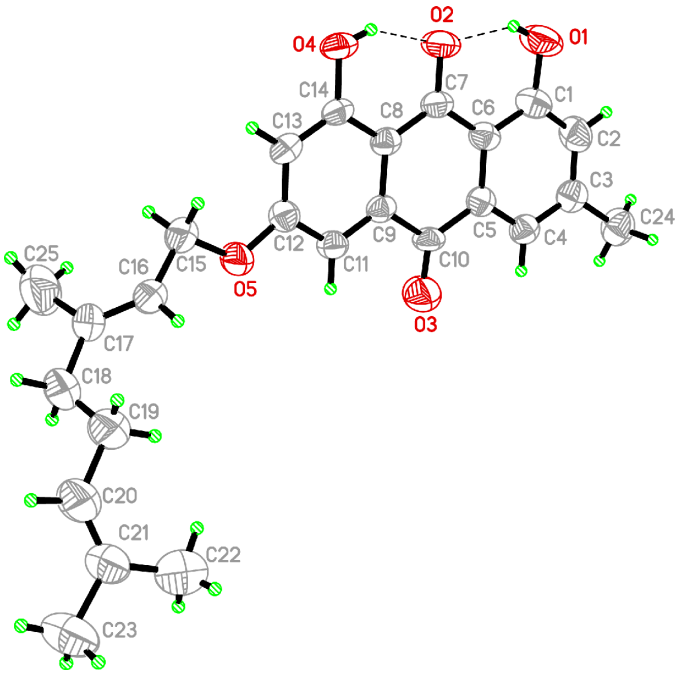


Figure 1
The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate hydrogen bonds.

interactions, generate $R_2^2(10)$ ring motifs (Bernstein *et al.*, 1995). The crystal packing is stabilized by weak π–π interaction between the anthraquinone ring system of the molecules translated by a unit along the *a* axis (Fig. 2). The ring system is stacked in such a way that the centroid–centroid distance between the C5–C10 ring at (*x*, *y*, *z*) and the C8–C13 ring at (1 + *x*, *y*, *z*) is 3.759 (2) Å, while the centroid–centroid separation between the C1–C6) ring at (*x*, *y*, *z*) and the C5–C10 ring at (*x* − 1, *y*, *z*) is 3.829 (2) Å.

Experimental

The air-dried bark of *Cratoxylum formosum ssp. pruniflorum* (4 kg) was ground and extracted with hexane and CH_2Cl_2 (2 × 20 l for each solvent for 5 d) at room temperature. The residue obtained after evaporation of the solvent was subjected to quick column chromatography (QCC) over silica gel and eluted with a gradient of hexane–ethyl acetate to afford 10 fractions (F1–F10). Fraction F2 (58.06 g) was further purified by QCC using a gradient of hexane–ethyl acetate as eluent to afford 25 fractions (17A–17Y). Fraction 17I was recrystallized from hexane–ethyl acetate (7:3 *v*/*v*) to give needle-shaped orange single crystals (m.p. 391–392 K; yield *ca* 0.10%).

Crystal data

$C_{25}H_{26}O_5$
M_r = 406.46
Triclinic, $P\overline{1}$
a = 4.6082 (1) Å
b = 12.965 (4) Å
c = 18.105 (5) Å
α = 100.074 (5)°
β = 92.978 (5)°
γ = 96.526 (5)°
V = 1055.3 (4) Å^3
Z = 2
D_x = 1.279 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1719 reflections
θ = 1.0–28.1°
μ = 0.09 mm^{-1}
T = 293 (2) K
Needle, orange
0.28 × 0.05 × 0.05 mm

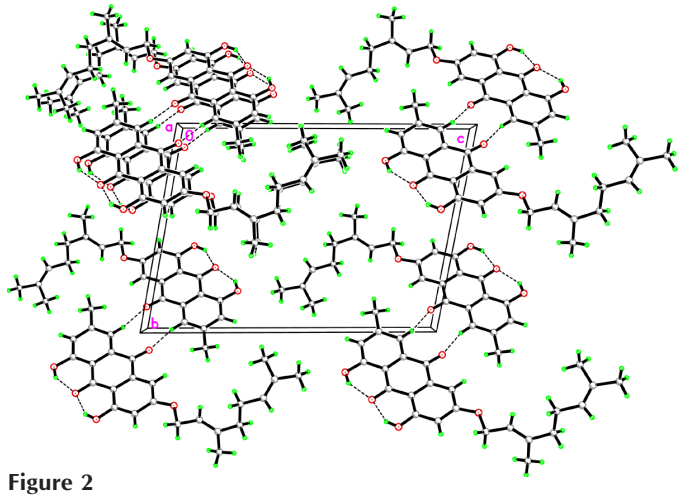


Figure 2
The packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.995, T_{max} = 0.996
9919 measured reflections
3693 independent reflections
2097 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 25.0°
h = −5 → 5
k = −15 → 15
l = −21 → 21

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.086
$wR(F^2)$ = 0.219
S = 1.07
3693 reflections
275 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0885P)^2 + 0.4977P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.346 (4)	O5—C12	1.353 (4)
O2—C7	1.260 (4)	O5—C15	1.446 (4)
O3—C10	1.223 (4)	C16—C17	1.314 (5)
O4—C14	1.346 (3)	C20—C21	1.299 (6)
C15—O5—C12—C11	178.7 (3)	C16—C17—C18—C19	1.1 (7)
C12—O5—C15—C16	−161.6 (3)	C17—C18—C19—C20	−175.7 (4)
O5—C15—C16—C17	−114.2 (4)	C18—C19—C20—C21	126.5 (6)
C15—C16—C17—C18	177.9 (4)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	1.86	2.583 (4)	146
O4—H4···O2	0.82	1.84	2.563 (3)	146
C4—H4*A*···O3^{i}	0.93	2.54	3.393 (5)	153

Symmetry code: (i) −2 − *x*, −*y*, −*z*.

H atoms were placed in calculated positions, with O—H distances of 0.82 Å and C—H distances in the range 0.93–0.97 Å. The U_{iso}

values were constrained to be $1.5U_{eq}$ of the carrier atom for hydroxyl and methyl H atoms and $1.2U_{eq}$ for the remaining H atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); 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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