2-(3,7-Dimethylocta-2,6-dienyl)-1,3,5,8-tetrahydroxyxanthone

M. Iqbal Choudhary,[a] Rasheeda Swaleh,[a] Shazia Anjum,[a]* Alain Meli Lannang,[b] Shamsher Ali[a] and Hoong-Kun Fun[c]*

[a]HEJ Research Institute of Chemistry, International Centre for Chemical Sciences, University of Karachi, Karachi-75270, Pakistan, [b]Department of Organic Chemistry, Faculty of Science, University of Yaounde I, PO Box 812, Cameroon, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

Correspondence e-mail:
anjumshazia@yahoo.com, hkfun@usm.my

Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.165
Data-to-parameter ratio = 12.0

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

The title compound, $C_{23}H_{24}O_6$, a xanthone derivative, was isolated from *Garcinia polyantha* Oliver. The orientation of the 3,7-dimethylocta-2,6-dienyl substituent with respect to the xanthone ring system is (+)synclinal. The crystal packing is stabilized by O—H⋯O and C—H⋯O intermolecular hydrogen bonds and π–π interactions.

Comment

Garcinia polyantha Oliver (family Guttiferae), also known as false chew stick tree, is found in West Africa, where its resinous sap is utilized in folk medicines for preparing wound dressings (Dalziel, 1937). The genus *Garcinia* is known to contain the pharmacologically active xanthones as secondary metabolites. This important class of compounds exhibits a variety of activities, such as antimicrobial (Malet-Cascon *et al.*, 2003), antifungal, monoamine-oxidase-inhibitory (Rocha *et al.*, 1994), anti-oxidant (Minami *et al.*, 1994), anti-inflammatory (Lin *et al.*, 1996) and antitumour (Ho *et al.*, 2002; Nkengfack *et al.*, 2002) activities. The oxygenated xanthones are reported to have antimalarial (Kelly *et al.*, 2002) and anti-HIV activities (Groweiss *et al.*, 2000). This paper presents the single-crystal X-ray structure analysis of the title compound, (I), a known compound (Komguem *et al.*, 2005) isolated from *Garcinia polyantha*. Compound (I) is also reported to possess modest antibacterial activity (Komguem *et al.*, 2005).

(I)
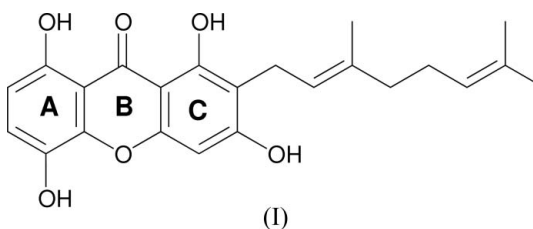
The xanthone ring system of (I) is planar, with a maximum deviation of 0.085 (2) Å for atom C3. The hydroxyl atoms O1, O2, O3 and O4 deviate from the xanthone mean plane by −0.046 (2), 0.202 (2), −0.023 (2) and 0.082 (2) Å, respectively. There is electron delocalization in rings *A* and *C* of the xanthone moiety and, as a result, the C8—C8*A* [1.408 (3) Å] and C1—C9*A* [1.421 (3) Å] bonds are longer than the C7—C8 [1.374 (3) Å], C7—C6 [1.384 (3) Å], C6—C5 [1.377 (3) Å], C1—C2 [1.383 (3) Å], C2—C3 [1.405 (3) Å], C3—C4 [1.385 (3) Å] and C4—C4*A* [1.368 (3) Å] bonds. The C3′—C2′—C1′ [127.4 (2)°] and C7′—C6′—C5′ [128.8 (2)°] bond angles in the 3,7-dimethylocta-2,6-dienyl substituent deviate
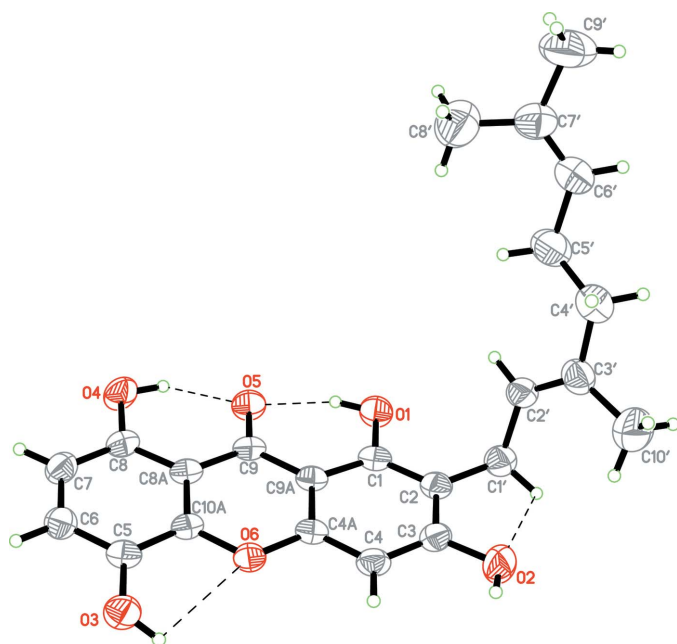
Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate hydrogen bonds.

Figure 2
The crystal packing of (I), showing O—H···O hydrogen-bonded (dashed lines) dimers.

significantly from $120°$. This may be as a result of H10A···H1'A [2.01 Å] and H8'A···H5'A [2.05 Å] close contacts. The 3,7-dimethylocta-2,6-dienyl substituent is attached to the xanthone ring system at C2, with C1—C2—C1'—C2' = 75.0 (2)°, indicating a (+)synclinal conformation.

As shown in Fig. 1, the C1'A—H1'A···O2 and O3—H1O3···O6 interactions generate rings of graph-set motif $S(5)$, while O1—H1O1···O5 and O4—H1O4···O5 interactions generate rings of graph-set motif $S(6)$ (Bernstein et al., 1995). In the crystal structure, the molecules form O2—H1O2···O3^i hydrogen-bonded centrosymmetric dimers [with an $R_2^2(18)$ ring motif] (Fig. 2), which are inter-linked by O3—H1O3···O4^{ii} and C4—H4A···O5^{ii} hydrogen bonds into a chain along the b cell axis (see Table 1 for symmetry codes). In addition, the crystal structure is stabilized by π–π interactions between inversion-related xanthone ring systems stacked along the a axis. The centroid···centroid distances between the rings are $Cg1···Cg1^{iii}$ = 3.523 (1) Å, $Cg1···Cg1^{iv}$ = 3.649 (1) Å, $Cg1···Cg3^{iv}$ = 3.686 (1) Å, $Cg2···Cg3^{iii}$ = 3.652 (1) Å and $Cg2···Cg3^{iv}$ = 3.558 (1) Å [symmetry codes: (iii) $-x, 1 - y, -z$; (iv) $1 - x, 1 - y, -z$], where $Cg1$, $Cg2$ and $Cg3$ are the centroids of the rings O6/C4A/C9A/C9/C8A/C10A, C1–C3/C4/C4A/C9A and C5–C8/C8A/C10A, respectively.

Experimental

Air-dried and pulverized twigs of Garcinia polyantha Oliver (2 kg), collected from Mount Kala, Cameroon, were extracted exhaustively with m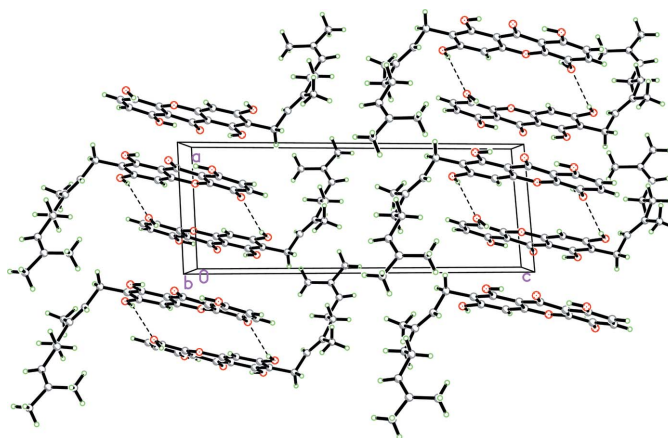ethanol (10 l). The combined extract was dried under reduced pressure and a gummy residue (223.7 g) was obtained. The crude methanolic extract was fractionated into hexane, dichloromethane, ethyl acetate and n-butanol extracts. The hexane extract (23.6 g) was fractionated over a silica-gel column with a hexane–dichloromethane and dichloromethane–methanol gradient. The fractions obtained at 1–2% methanol–dichloromethane, being similar, were pooled together. The yellow solid which settled at the bottom of the vessel was separated as a residue by filtering off the hexane-soluble portion. This residue (29.4 mg) was subjected to column chromatography on silica gel using dichloromethane as eluent. The pure compound was isolated as a lemon-yellow solid which, upon recrystallization from a mixture of dichloromethane and methanol (9.9:0.1), gave yellow plate-like crystals of (I) (14.6 mg, m.p. 489–491 K).

Crystal data

$C_{23}H_{24}O_6$	$Z = 2$
$M_r = 396.42$	$D_x = 1.364$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.1217$ (13) Å	Cell parameters from 6315
$b = 7.3672$ (14) Å	reflections
$c = 18.959$ (3) Å	$\theta = 1.1$–25.0°
$\alpha = 98.319$ (3)°	$\mu = 0.10$ mm^{-1}
$\beta = 92.196$ (3)°	$T = 293$ (2) K
$\gamma = 100.566$ (3)°	Plate, yellow
$V = 965.4$ (3) Å^3	$0.38 \times 0.18 \times 0.07$ mm

Data collection

Siemens SMART CCD area-detector diffractometer	3387 independent reflections
ω scans	2794 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{int} = 0.019$
	$\theta_{max} = 25.0°$
$T_{min} = 0.964$, $T_{max} = 0.993$	$h = -8 \rightarrow 8$
	$k = -8 \rightarrow 8$
9351 measured reflections	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0942P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.056$	$+ 0.2431P]$
$wR(F^2) = 0.165$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} = 0.006$
3387 reflections	$\Delta\rho_{max} = 0.58$ e Å^{-3}
282 parameters	$\Delta\rho_{min} = -0.41$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	Extinction correction: SHELXTL (Sheldrick, 1997)
	Extinction coefficient: 0.043 (5)

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
O1—H1O1⋯O5	0.87 (3)	1.80 (3)	2.612 (2)	153 (3)
O2—H1O2⋯O3^i	0.75 (4)	2.48 (4)	3.124 (2)	144 (3)
O3—H1O3⋯O4^{ii}	0.85 (3)	2.03 (3)	2.855 (2)	164 (3)
O3—H1O3⋯O6	0.85 (3)	2.38 (3)	2.729 (2)	105 (2)
O4—H1O4⋯O5	0.89 (3)	1.78 (3)	2.607 (2)	153 (3)
C1′—H1′A⋯O2	0.97	2.42	2.826 (3)	105
C4—H4A⋯O5^{ii}	0.93	2.49	3.200 (3)	133

Symmetry codes: (i) $-x + 1, -y + 2, -z$; (ii) $x, y + 1, z$.

Hydroxyl H atoms were located in a difference map and refined isotropically. All other H atoms were positioned geometrically [C—H = 0.93–0.97 Å] and allowed to ride on the parent atoms, with $U_{\text{iso}}(\text{H})$ = $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ for others. A rotating group model was used for the methyl groups.

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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