Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,6-Dihydroxy-3,7-dimethoxy-2,8-bis(3-methyl-2-butenyl)-9*H*-xanthen-9-one

Suchada Chantrapromma,[a]* Nawong Boonnak,[a] Hoong-Kun Fun[b]* and Chatchanok Karalai[c]

[a]Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [c]Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand

Correspondence e-mail: suchada.c@psu.ac.th, hkfun@usm.my

Received 13 December 2005
Accepted 19 December 2005

Key indicators

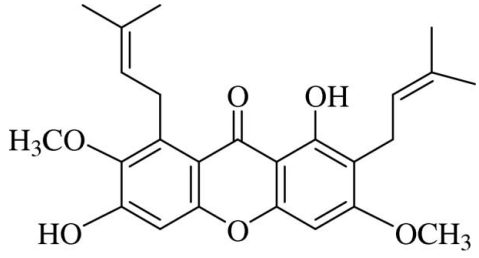
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.140
Data-to-parameter ratio = 19.7

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

The title compound, $C_{25}H_{28}O_6$, β-mangostin, is a xanthone which was isolated from *Cratoxylum formosum* ssp. *pruniforum*. O—H···O and C—H···O intramolecular hydrogen bonds are observed in the molecular structure. In the crystal packing, inversion-related molecules are stacked along the *a* axis with C—H···π and π–π interactions.

Comment

In our search for bioactive compounds from medicinal plants, we have investigated *Cratoxylum formosum* ssp. *pruniflorum*, which has been used for traditional medicine in Southeast Asia (Usher *et al.*, 1984). The main components of this plant are xanthones and anthraquinones. We have previously reported the crystal structures of an anthraquinone and xanthones which were isolated from this plant, *viz* 3-*O*-(geranyl)anthraquinone (Boonnak, Chantrapromma, Fun, Anjum *et al.*, 2005), xanthone V_1 (Chantrapromma *et al.*, 2005), prunifloxanthone A (Boonnak, Chantrapromma, Fun & Karalai, 2005) and macluraxanthone (Fun *et al.*, 2006). The title compound, (I), β-mangostin, is another xanthone which is a secondary metabolite occurring in this plant. It has previously been isolated from *Garcinia mangostana* (Asai *et al.*, 1995) and *Cratoxylum cochinchinense* (Nguyen & Harrison, 1999). Compound (I) has exhibited cytotoxicity against human leukemia HL 60 cells (Matsumoto *et al.*, 2003) and has antiproliferative effects against human colon cancer DLD-1 cells (Matsumoto *et al.*, 2005).



(I)

The present single-crystal structure determination of (I) is part of our ongoing search for bioactive compounds from Thai medicinal plants (Chantrapromma *et al.*, 2003, 2004, 2005; Boonnak, Chantrapromma, Fun, Anjum *et al.*, 2005; Boonnak, Chantrapromma, Fun & Karalai, 2005; Fun *et al.*, 2005, 2006). The structure-activity relationship (SAR) of xanthone derivatives will be investigated further.

The molecular structure of (I) is shown in Fig. 1, and selected bond distances and angles are given in Table 1. The

© 2006 International Union of Crystallography
All rights reserved

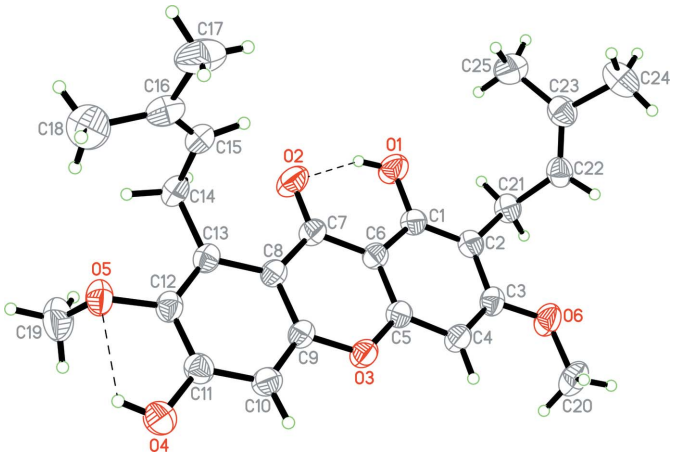


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Dashed lines indicate hydrogen bonds.

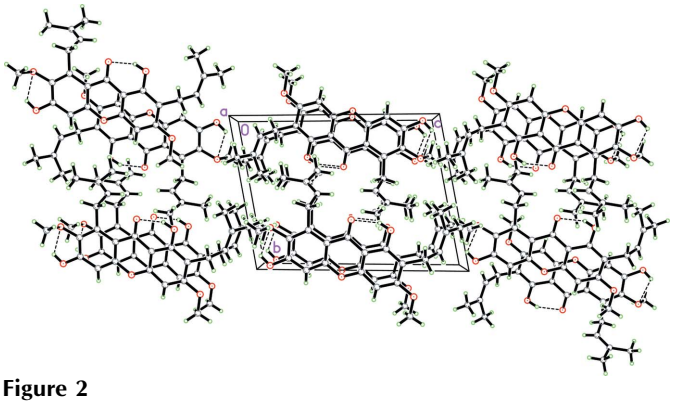


Figure 2
The crystal packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

bond distances and angles show normal values (Allen *et al.*, 1987) and are comparable with those in closely related structures (Chantrapromma *et al.*, 2005; Fun *et al.*, 2006).

The xanthene ring system of (I) (C1–C13/O3) is almost planar, with all atoms lying within 0.088 (1) Å of the mean plane. The dihedral angle between the two benzene rings of xanthene is 3.97 (5)°. The two hydroxyl groups are each coplanar with the attached rings. The methoxy group attached at atom C3 is coplanar with the xanthene ring system, with a C20—O6—C3—C2 torsion angle of 179.94 (11)°, while the other methoxy group attached at atom C12 is twisted away, with a C19–O5–C12–C11 torsion angle of −94.83 (13)°, indicating a (−)-anticlinal conformation (Fig. 1).

The two 3-methylbut-2-enyl substituents are attached to the xanthene ring system at C2 and C13; the torsion angles C1—C2—C21—C22 and C12—C13—C14—C15 are 100.42 (13) and 98.57 (12)°, respectively, both indicating a (+)-anticlinal conformation (Fig. 1). The attachment of the two 3-methylbut-2-enyl substituents is different from that observed in prunifloxanthone A (Boonnak, Chantrapromma, Fun & Karalai, 2005), in which one of them is in a (+)-anticlinal conformation and the other in a (−)-anticlinal conformation. This is due to the attachment of a methoxy group at atom C12 in (I) compared with a hydroxyl group attached at the same position in prunifloxanthone A. We expect that these differences would affect the bioactivities of these compounds.

O1—H1O1···O2 and O4—H1O4···O5 intramolecular hydrogen bonds generate *S*(6) and *S*(5) ring motifs, respectively (Bernstein *et al.*, 1995). There are also intramolecular C—H···O interactions present: C14—H14*A*···O2 generates an *S*(6) ring motif, C14—H14*B*···O5 generates an *S*(5) ring motif and C21—H21*B*···O1 generates an *S*(5) ring motif (Table 2). The crystal structure is stabilized by C—H···π interactions involving the C8–C13 benzene ring (centroid *Cg*1). The xanthene ring systems of inversion-related molecules are stacked in such a way that the centroids of the O3/C5–C9 ring at (*x*, *y*, *z*) and the C1–C6 benzene ring at (1 − *x*, −*y*, 1 − *z*) are 3.5697 (6) Å apart, indicating significant π–π interaction (Fig. 2).

Experimental

Air-dried roots of *C. formosum* ssp. *pruniflorum* (4 kg) were 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 over silica gel and eluted with a gradient of EtOAc–hexane to afford ten fractions (F1–F10). Fraction F2 was separated by column chromatography (CC) and eluted with 100% CH_2Cl_2 to afford four fractions (2A–2D). Fraction 2A was further purified by CC with 30% EtOAc–hexane to give compound (I). Compound (I) was recrystallized from $CHCl_3$–CH_3OH (4:1 *v*/*v*) to yield, after several days, yellow needle-shaped single crystals (m.p. 445–447 K).

Crystal data

$C_{25}H_{28}O_6$
M_r = 424.27
Triclinic, $P\overline{1}$
a = 7.8938 (1) Å
b = 10.1976 (1) Å
c = 13.7937 (2) Å
α = 79.311 (1)°
β = 80.926 (1)°
γ = 87.850 (1)°
V = 1077.40 (2) Å^3
Z = 2
D_x = 1.309 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 5720 reflections
θ = 1.5–29.0°
μ = 0.09 mm^{-1}
T = 100.0 (1) K
Block, yellow
0.50 × 0.28 × 0.23 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.969, T_{max} = 0.979
16706 measured reflections
5720 independent reflections
4623 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 29.0°
h = −10 → 10
k = −13 → 13
l = −18 → 18

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.140
S = 1.06
5720 reflections
291 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2 + 0.1769P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.3489 (13)	O5—C19	1.4348 (18)
O2—C7	1.2488 (13)	O6—C3	1.3602 (13)
O3—C5	1.3663 (13)	O6—C20	1.4217 (16)
O3—C9	1.3670 (12)	C15—C16	1.3274 (18)
O4—C11	1.3566 (14)	C22—C23	1.3316 (18)
O5—C12	1.3881 (13)		
C12—O5—C19	112.64 (10)	C3—O6—C20	118.14 (10)
O1—C1—C2—C3	−178.93 (10)	C19—O5—C12—C13	87.73 (14)
C20—O6—C3—C4	−0.80 (18)	O4—C11—C12—C13	177.68 (11)
C21—C2—C3—C4	179.16 (10)	C11—C12—C13—C14	−168.74 (10)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O1···O2	0.82	1.82	2.5558 (13)	148
O4—H1O4···O5	0.85 (2)	2.21 (2)	2.6994 (14)	117 (2)
C14—H14*A*···O2	0.97	2.27	2.8283 (15)	116
C14—H14*B*···O5	0.97	2.43	2.8573 (15)	106
C18—H18*A*···O5	0.96	2.59	3.413 (2)	144
C21—H21*B*···O1	0.97	2.40	2.8103 (15)	105
C20—H20*C*···*Cg*1^{i}	0.96	2.84	3.6661 (16)	144
C21—H21*A*···*Cg*1^{ii}	0.97	2.84	3.5627 (13)	132

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

Atom H1O4 was located in a difference map and refined isotropically. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with O—H = 0.82 Å and C—H = 0.93–0.97 Å. The U_{iso}(H) 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: *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).

The authors thank the Directed Basic Research in Medicinal Chemistry (Thailand Research Fund), the Prince of Songkla University, the Malaysian Government, and Universiti Sains Malaysia for Scientific Advancement Grant Allocation (SAGA) No. 304/PFIZIK/653003/A118. NW thanks the Development and Promotion of Science and Technology Talents Project and the Higher Education Development Project – Postgraduate Education and Research Programme in Chemistry for graduate study grants.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Asai, F., Tosa, H., Tanaka, T. & Iinuma M. (1995). *Phytochemistry*, **39**, 943–944.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Boonnak, N., Chantrapromma, S., Fun, H.-K., Anjum, S., Ali, S., Atta-ur-Rahman & Karalai, C. (2005). *Acta Cryst.* E**61**, o410–o412.
Boonnak, N., Chantrapromma, S., Fun, H.-K. & Karalai, C. (2005). *Acta Cryst.* E**61**, o4376–o4378.
Bruker (2005). *APEX2* (Version 1.27), *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chantrapromma, K., Saewan, N., Fun, H.-K., Chantrapromma, S. & Rahman, A. A. (2004). *Acta Cryst.* E**60**, o312–o314.
Chantrapromma, S., Boonnak, N., Fun, H.-K., Anjum, S. & Atta-ur-Rahman (2005). *Acta Cryst.* E**61**, o2136–o2138.
Chantrapromma, S., Fun, H.-K., Razak, I. A., Laphookhieo, S. & Karalai, C. (2003). *Acta Cryst.* E**59**, o1864–o1866.
Fun, H.-K., Ng, S.-L., Razak, I. A., Boonnak, N. & Chantrapromma, S. (2006). *Acta Cryst.* E**62**, o130–o132.
Fun, H.-K., Razak, I. A., Boonnak, N., Laphookhieo, S. & Chantrapromma, S. (2005). *Acta Cryst.* E**61**, o3086–o3088.
Matsumoto, K., Akao, Y., Kobayashi, E., Ohguchi, K., Ito, T., Tanaka, T., Iinuma, M. & Nozawa, Y. (2003). *J. Nat. Prod.* **66**, 1124–1127.
Matsumoto, K., Akao, Y., Ohguchi, K., Ito, T., Tanaka, T., Iinuma, M. & Nozawa, Y. (2005). *Bioorg. Med. Chem.* **13**, 6064–6069.
Nguyen, L. H. D. & Harrison, L. J. (1999). *Phytochemistry*, **50**, 471–476.
Sheldrick, G. M. (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Usher, G. (1984). *A Dictionary of Plants*, p.782. Delhi: CBS Publishers and Contributors.