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Key indicators

Single-crystal X-ray study
T = 100 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.055
wR factor = 0.145
Data-to-parameter ratio = 16.7

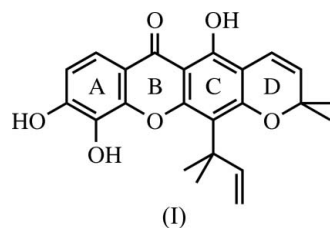
For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

12-(1,1-Dimethyl-2-propenyl)-5,9,10-trihydroxy-2,2-dimethyl-2H,6H-pyrano[3,2-b]xanthen-6-one

In the title compound, $\text{C}_{23}\text{H}_{22}\text{O}_6$, the xanthen ring system is essentially planar and the chromene ring is in a screw-boat conformation. Position 1 of the 1,1-dimethyl-2-propenyl substituent is coplanar with the attached ring. $\text{O}-\text{H}\cdots\text{O}$ intramolecular hydrogen bonds are observed in the structure. The molecules form centrosymmetric hydrogen-bonded dimers *via* weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions. The molecules are linked by intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds to form a one-dimensional chain along [010].

Comment

We have previously reported the crystal structures of a xanthone and a modified rotenoid containing the chromene ring, *viz* 5,9,10-trihydroxy-2,2-dimethyl-12-(3-methylbut-2-enyl)-2H,6H-pyrano[3,2-b]xanthen-6-one methanol solvate (Chantrapromma, Boonnak *et al.*, 2005) and 7a-O-methyldeguelol (Chantrapromma, Fun *et al.*, 2005). The title compound, (I), is another xanthone containing a chromene ring; since it also crystallized in the centrosymmetric space group $P2_1/c$, this indicates that (I) had been produced by non-enzymatic cyclization of a side chain (Chantrapromma, Boonnak *et al.*, 2005; Chantrapromma, Fun *et al.*, 2005).



Compound (I), macluraxanthone, was isolated from the bark of *Cratoxylum formosum* ssp. *prunifolium*, a shrub that was collected from Nhonkhai province in the northeastern part of Thailand. In our continuing search for bioactive compounds obtained from Thai medicinal plants (Chantrapromma *et al.*, 2004; Chantrapromma, Boonnak *et al.*, 2005; Chantrapromma, Fun *et al.*, 2005; Boonnak *et al.*, 2005; Fun *et al.*, 2005; Boonsri *et al.*, 2005), we have investigated *C. formosum* ssp. *pruniflorum*. Compound (I) has been reported previously (Monache *et al.*, 1981; Menache *et al.*, 1983; Goh *et al.*, 1992), but its X-ray crystal structure has not yet been reported.

The molecular structure of (I) is shown in Fig. 1. The bond distances and angles show normal values (Allen *et al.*, 1987) and are comparable to those in related structures (Chantrapromma & Boonnak *et al.*, 2005; Ravikumar *et al.*, 1987; Doriguetto *et al.*, 2001).

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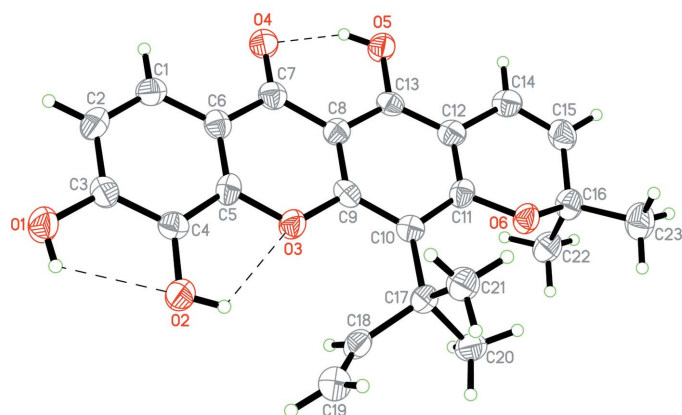


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The dashed lines indicate hydrogen bonds.

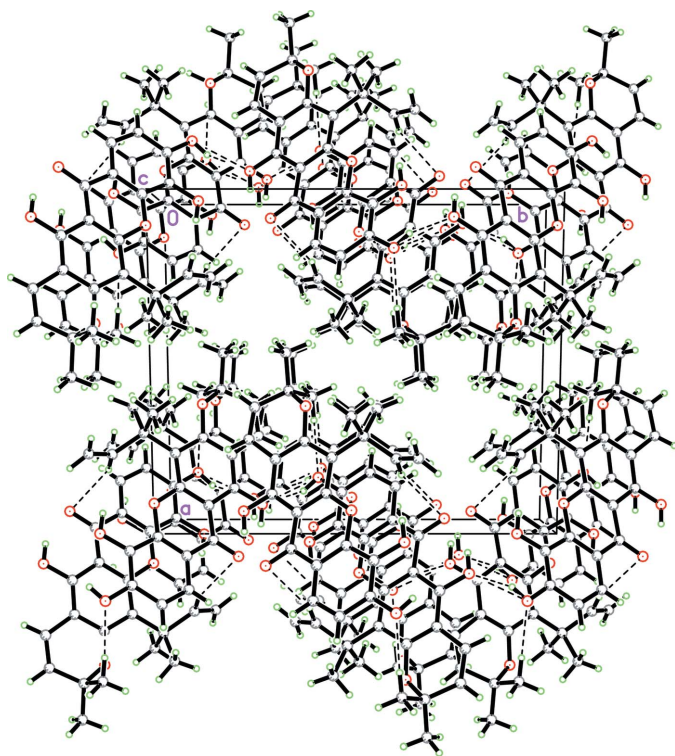


Figure 2
The crystal packing of (I), viewed down the *c* axis. Hydrogen bonds are shown as dashed lines.

The xanthene ring system (C1–C13/O3) is almost planar, with all atoms lying within 0.066 (2) Å of the mean plane. The three individual rings of xanthene are each essentially planar, the largest deviations from the ring planes being 0.010 (2), 0.033 (2) and 0.022 (2) Å for atoms C5, C6 and C9, C10 of rings *A*, *B*, and *C*, respectively. The dihedral angle between rings *A* and *B* is 2.60 (9)°, rings *B* and *C* form a dihedral angle of 5.73 (9)°, and the dihedral angle between rings *C* and *A* is 7.77 (9)°. The chromene ring, *D*, adopts a screw-boat conformation (Cremer & Pople, 1975), with puckering parameters $Q = 0.348$ (2) Å, $\theta = 64.0$ (3)° and $\varphi_2 = 326.7$ (4)°. The three hydroxyl groups are each coplanar with the attached rings. Atom C17 of the 1,1-dimethyl-2-propenyl substituent is

coplanar with ring *C*; the torsion angle C8–C9–C10–C17 is 176.93 (18)°.

In the crystal structure, there are intermolecular O1–H1O1···O5(–*x*, $\frac{1}{2}$ + *y*, $-\frac{1}{2}$ – *z*), C18–H18A···O4(–*x*, 2 – *y*, –*z*) and C23–H23A···O1(–*x*, 2 – *y*, –*z*) hydrogen bonds (Table 1). The molecules are linked by O–H···O intermolecular hydrogen bonds to form infinite chains along the *b* axis (Fig. 2). These chains form layers approximately parallel to the *bc* plane and are interconnected by the C–H···O interactions.

Experimental

Air-dried barks of *C. formosum* ssp. *prunifolium* (4 kg) were ground and extracted with hexane and CH₂Cl₂ (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 10 fractions (F1–F10). Fraction F3 was separated by column chromatography and eluted with 10% acetone–hexane to afford four fractions (3A–3D). Fraction 3C was recrystallized from CHCl₃–CH₃OH (4:1 v/v) to yield, after several days, yellow needle-shaped crystals of (I), suitable for single-crystal X-ray diffraction (m.p. 456–457 K).

Crystal data

C₂₃H₂₂O₆
M_r = 394.41
 Monoclinic, *P*2₁/*c*
a = 14.0220 (8) Å
b = 16.1568 (8) Å
c = 8.4157 (4) Å
 β = 104.423 (4)°
V = 1846.49 (17) Å³
Z = 4

D_x = 1.419 Mg m^{–3}
 Mo K α radiation
 Cell parameters from 4452 reflections
 θ = 2.0–28.0°
 μ = 0.10 mm^{–1}
T = 100.0 (1) K
 Needle, yellow
 0.46 × 0.08 × 0.04 mm

Data collection

Bruker SMART APEX-2 CCD
 area-detector diffractometer
 ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
T_{min} = 0.990, *T_{max}* = 0.996
 16825 measured reflections

4452 independent reflections
 2789 reflections with $I > 2\sigma(I)$
R_{int} = 0.076
 θ_{\max} = 28.0°
h = –18 → 18
k = –21 → 20
l = –11 → 11

Refinement

Refinement on *F*²
R [*F*² > 2 σ (*F*²)] = 0.055
wR(*F*²) = 0.145
S = 1.05
 4452 reflections
 266 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0623P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.34$ e Å^{–3}
 $\Delta\rho_{\min} = -0.25$ e Å^{–3}

Table 1
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>
O1–H1O1···O2	0.82	2.30	2.735 (2)	114
O1–H1O1···O5 ⁱ	0.82	1.97	2.720 (2)	153
O2–H1O2···O3	0.82	2.32	2.737 (2)	112
O5–H1O5···O4	0.82	1.77	2.509 (2)	150
C18–H18A···O4 ⁱⁱ	0.93	2.56	3.387 (3)	149
C20–H20B···O6	0.96	2.25	2.792 (3)	115
C23–H23A···O1 ⁱⁱ	0.96	2.56	3.492 (3)	164

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

H atoms were placed in calculated positions, with O–H distances of 0.82 Å and C–H distances in the range 0.93–0.96 Å. The $U_{\text{iso}}(\text{H})$ 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 and cell refinement: *APEX2* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); structure solution: *SHELXTL* (Sheldrick, 1998); structure refinement: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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