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5,9,10-Trihydroxy-2,2-dimethyl-12-(3-methylbut-2-enyl)-2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one methanol solvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.087
wR factor = 0.230
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.

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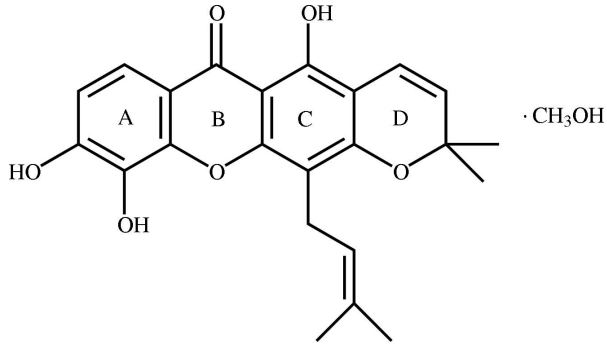
The title compound, $C_{23}H_{22}O_6\cdot CH_3OH$, has an essentially planar xanthone nucleus fused to a chromene ring which adopts a sofa conformation. The 3-methylbut-2-enyl substituent is axially attached to the xanthone ring system, with a (+)-anticlinal conformation. The crystal structure is stabilized by O—H···O and C—H···O intramolecular and intermolecular interactions.

Comment

Cratoxylum is a small genus belonging to the Guttiferae family, with at least six species distributed in several Southeast Asian countries (Iinuma *et al.*, 1996). Species of this genus have been used for their diuretic, stomachic, and tonic effects (Kitanov *et al.*, 1988), as well as for diarrhea and flatulence (Anderson, 1986), and for food poisoning and internal bleeding (Grosvenor *et al.*, 1995). Some species of this genus exhibit antimalarial and antiprotozoal activity, and are slightly cytoxic against human L6 cells (Seo *et al.*, 2002; Zakaria, 2004). In our continuing search for bioactive compounds from Thai medicinal plants (Chantrapromma *et al.*, 2003; Boonnak *et al.*, 2005), we have investigated *Cratoxylum formosum* ssp. *pruniflorum*, a shrub which is known locally to Thais as Tuikhon. Tuikhon was collected from Nhongkhai province and is widely distributed in the north-eastern part of Thailand. We have isolated the title compound, (I), xanthone V_1, for the first time from *Cratoxylum formosum* ssp. *pruniflorum*. It was previously isolated from *Vismia guineensis* (Botta *et al.*, 1986) and *Garcinia latissima* (Ito *et al.*, 1997).



(I)

Because of the puckering of atoms O6 and C16, the title molecule is chiral, but crystallized in the centrosymmetric space group $P2_1/c$. This indicates that the crude extract from which the compound was obtained is a racemic mixture and that (I) was produced by non-enzymatic cyclization of a side chain. A closely related structure, having an identical skeleton, is dulxanthone E (Kosela *et al.*, 1999).

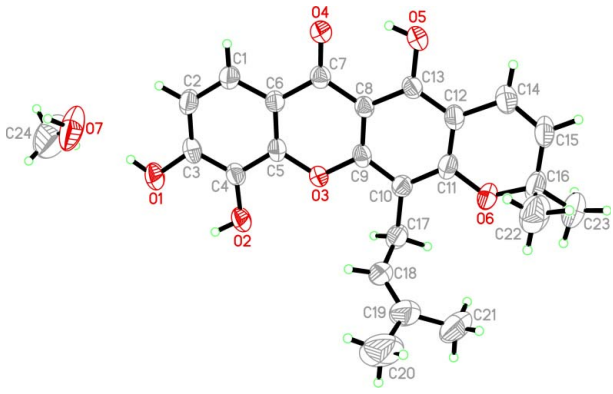


Figure 1
The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

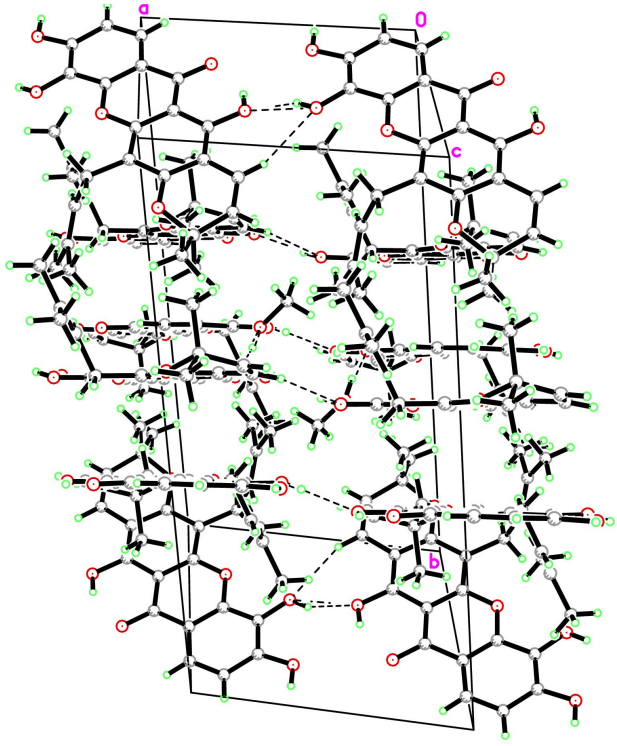


Figure 2
A view of the molecular packing. Dashed lines indicate hydrogen bonds.

The xanthone skeleton (rings *A*, *B* and C) is essentially planar, with a maximum deviation of 0.044 (2) Å for atom O3. The chromene ring *D* is in a sofa conformation, with puckering parameter Q = 0.297 (5) Å (Cremer & Pople, 1975), atom C16 having the maximum deviation of 0.190 (4) Å. The two methyl groups are axially and bisectionally attached to the chromene ring at atom C16, with torsion angles C14—C15—C16—C22 of −93.7 (6)° and C14—C15—C16—C23 of 141.8 (5)°. The 3-methylbut-2-enyl substituent is attached to ring C at C10, with C11—C10—C17—C18 = 97.4 (4)°, indicating a (+)-anticlinal conformation (Fig. 1).

The bond lengths and angles in (I) have normal values (Allen *et al.*, 1987) and are comparable to those in dulxanthone E (Kosela *et al.*, 1999). Intramolecular and intermolecular O—H···O and weak C—H···O interactions are observed (Table 2). The molecules are linked together by these interactions to form a three-dimensional molecular network (Fig. 2).

Experimental

Air-dried barks 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 (QCC) over silica gel and eluted with a gradient of EtOAc-hexane to afford ten fractions (F1–F10). Fraction F6 (3.72 g) was separated by column chromatography (CC), eluted with 15% EtOAc–hexane to afford seven fractions (6 A–6 G). Fraction 6B was further purified by CC with 30% EtOAc–hexane to give two fractions (6BA and 6BB). Fraction 6BA was recrystallized from $CHCl_3$–CH_3OH (8:2 *v*/*v*) to give brown single crystals of (I) after several days (m.p. 491–492 K).

Crystal data

$C_{23}H_{22}O_6·CH_4O$
M_r = 426.45
Monoclinic, $P2_1/c$
a = 9.9258 (11) Å
b = 20.089 (2) Å
c = 11.8605 (13) Å
β = 111.324 (2)°
V = 2203.1 (4) Å^3
Z = 4
D_x = 1.286 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11187 reflections
θ = 2.0–25.0°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, brown
0.66 × 0.29 × 0.13 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.968, T_{max} = 0.988
11187 measured reflections
3875 independent reflections
3309 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.0°
h = −11 → 11
k = −23 → 23
l = −14 → 6

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.087
$wR(F^2)$ = 0.230
S = 1.11
3875 reflections
289 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0696P)^2 + 5.0192P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.356 (4)	O5—C13	1.344 (5)
O2—C4	1.354 (5)	O6—C11	1.358 (5)
O3—C5	1.365 (4)	O6—C16	1.468 (5)
O3—C9	1.372 (4)	O7—C24	1.369 (8)
O4—C7	1.252 (4)	C18—C19	1.314 (7)
C5—O3—C9	119.3 (3)	O6—C16—C23	103.9 (4)
C11—O6—C16	119.1 (3)	C15—C16—C23	112.0 (4)
C9—C10—C17	121.6 (3)	O6—C16—C22	107.4 (4)
C11—C10—C17	122.4 (3)	C15—C16—C22	111.4 (5)
C14—C15—C16—C23	141.8 (5)	C11—C10—C17—C18	97.4 (4)
C14—C15—C16—C22	−93.7 (6)	C17—C18—C19—C20	−178.8 (7)
C9—C10—C17—C18	−81.4 (5)	C17—C18—C19—C21	−1.4 (11)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O7^{i}	0.82	1.85	2.668 (5)	171
O2—H2A···O1^{i}	0.82	2.31	2.718 (5)	111
O2—H2A···O5^{ii}	0.82	2.01	2.792 (5)	160
O5—H5A···O4^{i}	0.82	1.84	2.570 (4)	148
O7—H7A···O4^{iii}	0.82	1.97	2.789 (4)	171
C14—H14A···O2^{iv}	0.93	2.51	3.394 (6)	159
C17—H17B···O6^{i}	0.97	2.42	2.807 (6)	103

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

H atoms were placed in calculated positions with an O—H distance of 0.82 Å and C—H distances in the range 0.93–0.98 Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atoms for hydroxyl and methyl H atoms and $1.2U_{eq}$ for the other 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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