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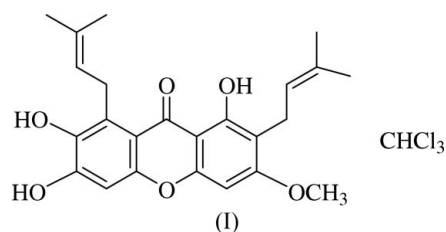
Key indicators

Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.063
 wR factor = 0.218
Data-to-parameter ratio = 13.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.1,6,7-Trihydroxy-3-methoxy-2,8-bis(3-methyl-
2-butenyl)-9*H*-xanthen-9-one chloroform solvate

The title compound, $\text{C}_{24}\text{H}_{26}\text{O}_6 \cdot \text{CHCl}_3$, which we named prunifloxanthone A, was isolated for the first time from the roots of *Cratoxylum formosum* ssp. *pruniflorum* and has never been isolated from any other natural resources as yet. The xanthone ring systems of the molecules are stacked along the a axis, with significant π - π interactions. In addition, $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds link glide-related molecules into a chain along [201].

Comment

We have previously reported the crystal structures of the compounds 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 (Chantrapromma *et al.*, 2005) and 3-(3,7-dimethylocta-2,6-dienyloxy)-1,8-dihydroxy-6-methyl-9,10-anthraquinone (Boonnak *et al.*, 2005) isolated from the bark of *Cratoxylum formosum* ssp. *pruniflorum* or 'Tuikhon' in Thai, a medicinal plant growing in the northeastern part of Thailand. Xanthenes are the main components of this plant. The title compound, (I), prunifloxanthone A, is a new xanthone which was for the first time isolated from *C. pruniflorum* and has never been isolated from any other natural resources as yet. Compound (I) is found to exhibit antioxidant activity. The present single-crystal structure determination of (I) is a part of our ongoing studies on the biological activities of Thai medicinal plants (Chantrapromma *et al.*, 2003, 2004, 2005; Boonnak *et al.*, 2005; Fun *et al.*, 2005). The structure-activity relationships (SAR) of these compounds will be further studied.



The bond lengths and angles in (I) (Table 1) show normal values (Allen *et al.*, 1987) and are comparable to those reported in a closely related structure (Chantrapromma *et al.*, 2005). The xanthone ring system is approximately planar (r.m.s. deviation 0.044 Å), with a maximum deviation of 0.093 (3) Å for atom C7. The methoxy group attached at atom C3 is almost coplanar with the xanthone ring system with a C19-O4-C3-C4 torsion angle of 2.8 (4)°. One of the 3-methylbut-2-enyl substituents is attached to the xanthone ring system at C2, with C1-C2-C20-C21 = 91.1 (4)°, indicating a (+)-anticlinal conformation (Fig. 1). The other 3-methylbut-

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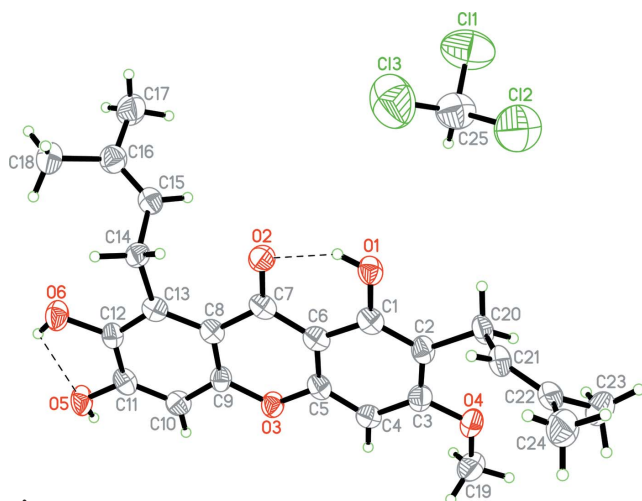


Figure 1
The structure of (I), showing 80% probability displacement ellipsoids and the atomic numbering. Dashed lines indicate O—H...O hydrogen bonds.

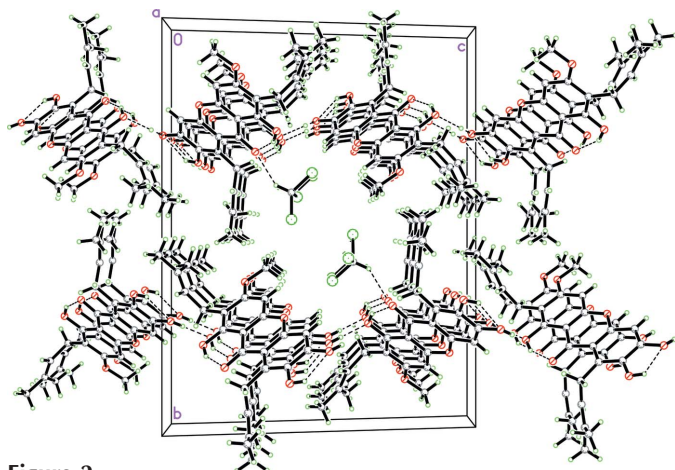


Figure 2
The crystal packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

2-enyl group is attached at C13, with C12—C13—C14—C15 = $-95.4(3)^\circ$, indicating a (–)-anticonformation (Fig. 1).

The O1—H1...O2 and O6—H6...O5 intramolecular hydrogen bonds (Table 2) generate *S*(6) and *S*(5) ring motifs, respectively (Bernstein, *et al.*, 1995). In addition, a weak C14—H14A...O6 interaction generates an *S*(5) ring motif and a C14—H14B...O2 interaction generates an *S*(6) ring motif.

The xanthone ring systems of the molecules are stacked along the *a* axis in such a way that the centroids of the C1—C6 and C8—C13 benzene rings are 3.557(2) Å apart, indicating significant π – π interactions (Fig. 2). In addition, glide-related molecules are linked into a chain along [201] via O5—H5...O1ⁱ hydrogen bonds (symmetry code in Table 2). The chloroform molecule is linked to the xanthone derivative via a C—H...O hydrogen bond (Table 2).

Experimental

Air-dried roots of *C. formosum* ssp. *pruniflorum* (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 (QCC) over silica gel and eluted with a gradient of EtOAc–hexane to afford ten fractions (F1–F10). Fraction F7 was separated by QCC and eluted with a gradient of acetone–hexane to afford seven fractions (7A–7G). Fraction 7C was further purified by column chromatography with 80% CH₂Cl₂–hexane to give four fractions (7CA–7CD). Fraction 7CA was recrystallized from CHCl₃–CH₃OH (7:3 v/v) to give yellow needle-shaped single crystals of (I) after several days (m.p. 395–397 K).

Crystal data

C₂₂H₂₆O₆·CHCl₃
M_r = 529.82
 Monoclinic, *P*2₁/*c*
a = 5.9059 (1) Å
b = 23.3103 (3) Å
c = 18.4042 (2) Å
 β = 103.438 (1)°
V = 2464.31 (6) Å³
Z = 4

D_x = 1.428 Mg m^{−3}
 Mo *K*α radiation
 Cell parameters from 4316 reflections
 θ = 1.4–26.0°
 μ = 0.41 mm^{−1}
T = 100.0 (1) K
 Block, yellow
 0.37 × 0.22 × 0.13 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
T_{min} = 0.897, *T_{max}* = 0.948
 38758 measured reflections

4316 independent reflections
 3713 reflections with *I* > 2σ(*I*)
R_{int} = 0.034
 θ_{\max} = 25.0°
h = −7 → 7
k = −27 → 27
l = −21 → 21

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.063
wR (*F*²) = 0.218
S = 1.08
 4316 reflections
 315 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1248P)^2 + 5.5318P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.79 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.74 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

C11—C25	1.753 (5)	O4—C3	1.364 (4)
C12—C25	1.746 (4)	O4—C19	1.424 (4)
C13—C25	1.758 (5)	O5—C11	1.356 (4)
O1—C1	1.365 (4)	O6—C12	1.372 (4)
O2—C7	1.257 (4)	C15—C16	1.323 (5)
O3—C5	1.364 (4)	C21—C22	1.326 (5)
O3—C9	1.383 (4)		
O3—C5—C4	116.0 (3)	O3—C9—C8	123.4 (3)
C4—C5—C6	123.5 (3)	C16—C15—C14	126.1 (3)
C5—C6—C1	116.4 (3)	C15—C16—C18	123.0 (3)
C5—C6—C7	121.7 (3)	C17—C16—C18	115.1 (3)
C9—C8—C7	118.0 (3)	C22—C21—C20	128.6 (3)
C13—C8—C7	123.7 (3)	C21—C22—C23	124.4 (3)
O3—C9—C10	113.6 (3)	C24—C22—C23	114.8 (3)

Table 2

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—H1...O2	0.82	1.77	2.505 (3)	149
O5—H5...O1 ⁱ	0.82	1.88	2.695 (3)	176
O6—H6...O5	0.82	2.18	2.643 (3)	116
C14—H14A...O6	0.97	2.36	2.769 (4)	105
C14—H14B...O2	0.97	2.31	2.825 (4)	112
C25—H25...O2 ⁱⁱ	0.98	2.42	3.256 (5)	142

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

H atoms were placed in calculated positions with O–H distances of 0.82 Å and C–H distances in the range 0.93–0.98 Å. The U_{iso} values were constrained to be $1.5U_{\text{eq}}$ of the carrier atoms for hydroxy and methyl H atoms, and $1.2U_{\text{eq}}$ for the remaining H atoms. A rotating group model was used for the hydroxy and methyl H atoms. The data were collected with an Oxford Cyrosystem Cobra low-temperature attachment.

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).

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