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90112, Thailand, and ^bX-ray Crystallography
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Key indicators

Single-crystal X-ray study
 $T = 297$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.055
 wR factor = 0.152
Data-to-parameter ratio = 14.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.4-(1,1-Dimethylprop-2-enyl)-1,3,5,6-tetrahydroxy-
2-(3-methylbut-2-enyl)-9H-xanthen-9-one mono-
hydrate

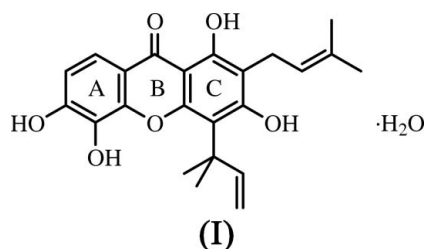
The title compound, $\text{C}_{23}\text{H}_{24}\text{O}_6 \cdot \text{H}_2\text{O}$, a xanthone compound, was isolated from *Cratoxylum formosum* ssp. *pruniflorum*. The three rings in the molecule are coplanar. The 3-methylbut-2-enyl and 1,1-dimethylprop-2-enyl side chains are axially attached to the benzene ring. There are intra- and intermolecular $\text{O}-\text{H} \cdots \text{O}$ and $\text{C}-\text{H} \cdots \text{O}$ interactions. The crystal structure is stabilized by these intermolecular interactions as well by $\text{C}-\text{H} \cdots \pi$ interactions.

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Comment

Cratoxylum is a small genus belonging to the Guttiferae family and is found mainly in southeast Asia (Robson, 1974). Some species of this genus have been used for the treatment of diuretic and stomachic symptoms, for tonic effects (Kitanov *et al.*, 1988), and for diarrhea and flatulence (Aderson 1986). These plants produce various types of secondary metabolites, including xanthenes (Kijjoa *et al.*, 1998), triterpenoids (Nguyen & Harrison, 1998; Bennett *et al.*, 1993) and flavonoids (Kitanov *et al.*, 1988). In our previous studies, we have reported a number of crystal structures of xanthone and anthraquinone from *C. formosum* ssp. *pruniflorum*, a medicinal plant growing in the northeastern part of Thailand (Boonnak, Chantrapromma, Fun, Anjum *et al.*, 2005; Boonnak, Chantrapromma, Fun & Karalai, 2005; Chantrapromma *et al.*, 2005). As a continuation of our study on this genus, the compound known as gerontoxanthone was isolated. We report here the structure of gerontoxanthone monohydrate, (I).



In (I), the xanthone skeleton (rings *A*, *B* and *C*) is almost planar (Fig. 1). Selected bond lengths and angles are given in Table 1. The bond distances and bond angles show normal values (Allen *et al.*, 1987), comparable to those observed in some closely related compounds (Boonnak, Chantrapromma, Fun & Karalai 2005; Chantrapromma *et al.*, 2005). The maximum deviation from planarity in the essentially planar skeleton (C1–C13/O3) is 0.015 (2) Å for atom C7. The 3-methylbut-2-enyl (C14–C18) side chain is axially attached to the benzene ring *C* and a C1–C2–C14–C15 torsion angle of

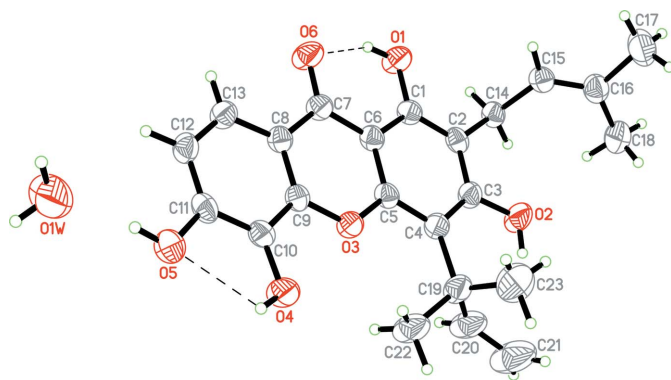


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate intramolecular hydrogen bonds.

90.6 (2)°, indicating a (+)-synclinal conformation, and the 1,1-dimethylprop-2-enyl (C19–C23) substituent is axially attached to ring C and a C3–C4–C19–C20 torsion angle of –52.6 (3)°, indicating a (–)-synclinal conformation.

Intramolecular and intermolecular O–H···O and C–H···O interactions are observed (Table 2). The water molecule and all hydroxy O atoms are involved in hydrogen bonds. The O1–H1···O6 and O4–H4···O5 intramolecular hydrogen bonds generate $R_1^1(6)$ and $R_1^1(5)$ motifs, respectively (Bernstein *et al.*, 1995). There is a weak intramolecular C–H···O interaction (C23–H23C···O2), which generates an $R_1^1(6)$ ring motif. There is also a C–H···Cg interaction between one of the methyl groups of the 3-methylbut-2-enyl side chain and the centroid Cg1 of ring A. The molecules are linked together to form a three-dimensional network (Fig. 2).

Experimental

Air-dried bark of *C. formosum* ssp. *prunforum* (4 kg) was ground and extracted with hexane and CH₂Cl₂ (20 l for each solvent) over a period of 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 ethyl acetate–hexane to afford 10 fractions (F1–F10). Fraction F5 was separated by column chromatography (CC) with 10% acetone–hexane to afford six fractions (5A–5F). Fraction 5D was purified by CC with 15% acetone–hexane to give three fractions (5D1–5D3). Fraction 5D2 was recrystallized from CHCl₃–CH₃OH (4:1 v/v) to give yellow plate-shaped single crystals of (I) after several days (m.p. 453–454 K).

Crystal data

C₂₃H₂₄O₆·H₂O
M_r = 414.44
 Monoclinic, $P2_1/c$
a = 10.0244 (6) Å
b = 19.7901 (11) Å
c = 11.7645 (5) Å
 β = 116.217 (4)°
V = 2093.8 (2) Å³

Z = 4
D_x = 1.315 Mg m^{–3}
 Mo *K*α radiation
 μ = 0.10 mm^{–1}
T = 297 (2) K
 Plate, yellow
 0.46 × 0.20 × 0.06 mm

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.749, *T_{max}* = 0.994

11308 measured reflections
 4101 independent reflections
 3319 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
 θ_{\max} = 26.0°

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.055
 $wR(F^2)$ = 0.152
S = 1.11
 4101 reflections
 288 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 0.7399P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.44 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.25 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1–C1	1.353 (2)	O5–C11	1.357 (2)
O2–C3	1.359 (2)	O6–C7	1.260 (2)
O3–C9	1.364 (2)	C15–C16	1.319 (3)
O3–C5	1.366 (2)	C20–C21	1.318 (4)
O4–C10	1.352 (2)		
C16–C15–C14	127.52 (19)	C21–C20–C19	127.9 (3)
C18–C16–C17	114.5 (2)		

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···O6	0.82	1.81	2.553 (2)	149
O4–H4···O1 ⁱ	0.82	2.04	2.805 (2)	156
O4–H4···O5	0.82	2.29	2.710 (2)	112
O5–H5···O1W	0.82	1.86	2.674 (3)	170
O1W–H2W1···O6 ⁱⁱ	0.83 (3)	1.97 (2)	2.797 (3)	172 (5)
C14–H14B···O4 ⁱⁱⁱ	0.97	2.50	3.364 (3)	148
C23–H23C···O2	0.96	2.57	3.167 (5)	121
C17–H17C···Cg1 ^{iv}	0.96	2.94	3.595 (3)	129

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

H atoms of the water molecule and that attached to O2 were located in difference maps. Restraints [$O1W-H1W1 = O1W-H1W2 = 0.82$ (1) Å] were applied to yield an ideal water molecule configuration. The remaining H atoms were placed in calculated positions, with an O–H distance of 0.82 Å and C–H distances in the range 0.93–0.97 Å. The U_{iso} values were constrained to be $1.5U_{\text{eq}}$ of the carrier atom for hydroxy and methyl H atoms and $1.2U_{\text{eq}}$ for the remaining 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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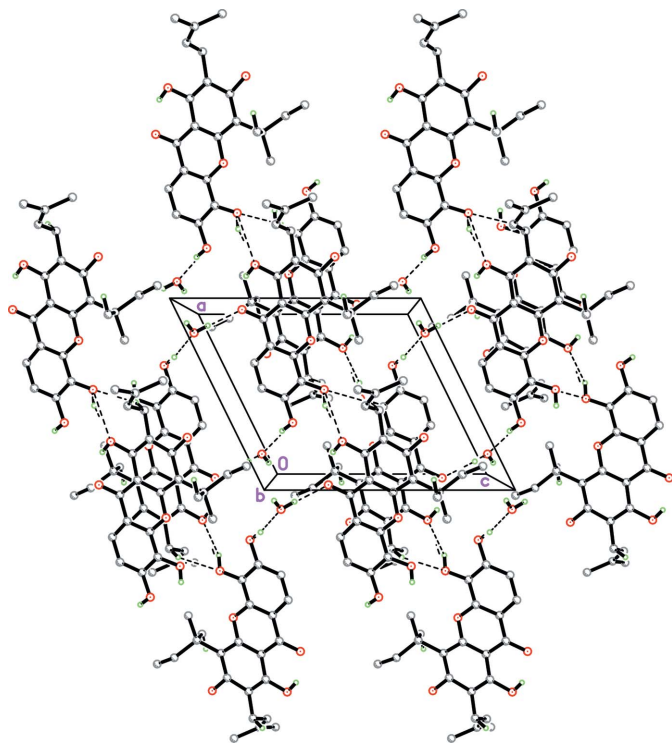


Figure 2
A view of the molecular packing down the *b* axis. H atoms have been omitted unless they are involved in hydrogen bonds (dashed lines).

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