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7-Glycoloyl-2-hydroxy-1,4b,7,10a-tetramethyl-4a,4b,5,6,7,8,8a,9,10,10a-decahydrophenanthren-3(4*H*)-one

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.134
Data-to-parameter ratio = 10.1

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

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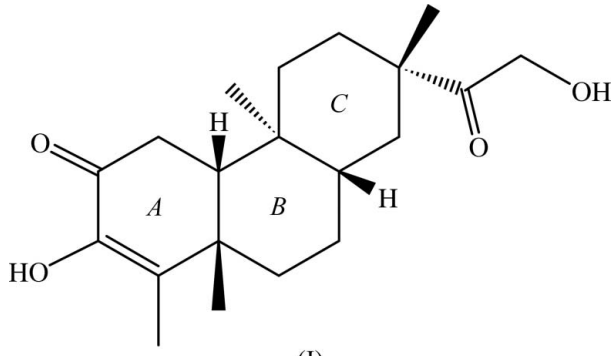
The title compound, $C_{20}H_{30}O_4$, is a diterpenoid which was isolated from the roots of *Ceriops tagal* (Perr.). The asymmetric unit consists of three crystallographically independent molecules, which are conformationally almost identical. The molecule has three fused rings in the structure; the cyclohexene ring *A* adopts a half chair conformation and the two cyclohexane rings *B* and *C* are in standard chair conformations. The molecular structure is stabilized by intra- and intermolecular O—H···O hydrogen bonds and weak C—H···O interactions. In the crystal structure, molecules are linked into infinite one-dimensional chains along the *b* axis.

Comment

Ceriops tagal (Perr.) C. B. Robinson is a mangrove plant belonging to the Rhizophoraceae family. It has been reported that its decoction has been used as a substitute for quinine in the treatment for malaria (Bamroongrugsa, 1999). In our continuing research on bioactive compounds from mangrove sources (Cheenpracha *et al.*, 2005; Pakhathirathien *et al.*, 2005; Chantrapromma *et al.*, 2006; Chumkaew *et al.*, 2006; Fun, Chantrapromma *et al.*, 2006; Fun, Pakhathirathien *et al.*, 2006; Koysomboon *et al.*, 2006), we have studied the chemical constituents from the roots and barks of *C. Tagal* (Perr.) to search for the antimalarial active components. We have previously reported crystal structures of two compounds which were isolated from this plant (Chantrapromma *et al.*, 2006; Fun, Pakhathirathien *et al.*, 2006). We report here the crystal structure of the title compound, (I), which was isolated from the roots of this plant. Compound (I) was not found to possess antimalarial activity compared with the artemisinin standard which has an IC_{50} value of 3.3–3.9 n*M*.



(I)

The title compound crystallized with three crystallographically independent molecules, *A*, *B* and *C*, in the asymmetric unit (Fig. 1; suffixes A, B and C of the atom labels

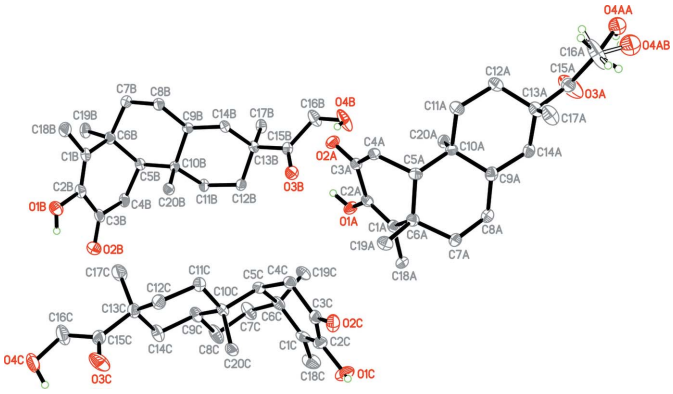


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atomic numbering. For clarity, only the hydroxyl and disordered H atoms have been shown.

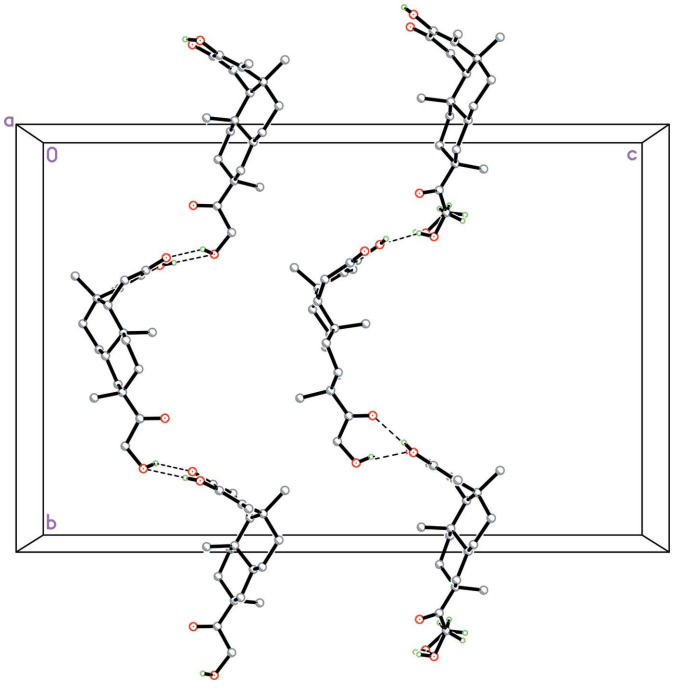


Figure 2
A partial packing diagram of (I), viewed down the *a* axis. The O—H···O hydrogen bonds are shown as dashed lines. H atoms, except for the hydroxyl and disordered H atoms, have been omitted for clarity.

correspond to molecules *A*, *B* and *C*, respectively), which are slightly different in bond lengths and angles from each other. The bond distances and angles in (I) are within normal ranges (Allen *et al.*, 1987) and comparable with those of a closely related structure (Fun, Pakhathirathien *et al.*, 2006). The molecule of (I) contains a fused three-ring system *A*/*B*/*C* (see scheme). The *A*/*B* ring junction is *cis*-fused and *B*/*C* is *trans*-fused. The cyclohexene ring *A* adopts a half-chair conformation with puckering parameters [Q = 0.426 (3) Å, θ = 129.3 (4)° and φ = 63.8 (5)° in molecule *A*; Q = 0.445 (3) Å, θ = 130.3 (4)° and φ = 66.9 (5)° in molecule *B*; Q = 0.444 (3) Å, θ = 127.5 (4)° and φ = 60.9 (5)° in molecule *C*] and rings *B* and *C* have standard chair conformations (Cremer & Pople 1975). The methyl and hydroxyl groups attached to cyclohexene ring *A* at atom C1 and C2, respectively, form a planar unit extending across atoms O1, O2, C1, C2, C3, C4 and C18 as indicated by the bond angles of *ca* 120° around C1, C2 and C3, indicative of sp^2-hybridization for these atoms. The intramolecular O1—H1···O2 hydrogen bond (Table 1) further contributes to the planarity of this section of the molecule. The orientation of the glycoloyl substituent [C15–C16/O3–O4] at C13 can be described by the torsion angle of C14—C13—C15—C16 [−149.6 (3), −91.4 (3) and −89.0 (4)° for molecule *A*, *B* and *C*, respectively]. The CH_2OH group of the glycoloyl substituent in molecule *A* is disordered over two sites (see Fig. 1).

The crystal packing of (I) is stabilized by intermolecular O—H···O hydrogen bonds (Table 1), forming chains along the *b* axis (Fig. 2), and further stabilized by weak C—H···O interactions.

Experimental

The air-dried and crushed roots of *C. tagal* (3.6 kg), which was collected from Nakhon Si Thammarat province in the southern part of Thailand, were extracted with dichloromethane and then concentrated *in vacuo* to give a residue (16.3 g). This residue was subjected to quick column chromatography over silica gel using solvents of increasing polarity from hexane to acetone/hexane (1:1 *v*/*v*). The eluates were collected and combined, based on TLC, to give 14 fractions (F1–F14). Fraction F14 was further purified by repeated quick column chromatography with 15% acetone in dichloromethane, yielding compound (I) (15.0 mg). Colorless plate crystals were obtained from recrystallization in hexane/dichloromethane (1:4 *v*/*v*) after several days [m.p. 425–429 K].

Crystal data

$C_{20}H_{30}O_4$
M_r = 334.44
Orthorhombic, $P2_12_12_1$
a = 7.8316 (4) Å
b = 21.0250 (9) Å
c = 32.0885 (13) Å
V = 5283.7 (4) Å^3
Z = 12
D_x = 1.261 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100.0 (1) K
Plate, colorless
0.44 × 0.40 × 0.08 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.963, T_{max} = 0.994
56698 measured reflections
6724 independent reflections
5787 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.134
S = 1.09
6724 reflections
665 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.068P)^2 + 2.2807P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1*A*—H1*AA*···O2*A*	0.82	2.28	2.707 (3)	113
O1*A*—H1*AA*···O3*B*	0.82	2.02	2.776 (3)	152
O4*AA*—H4*AA*···O1*B*^{i}	0.82	2.24	2.997 (5)	155
O4*AA*—H4*AA*···O3*A*	0.82	2.24	2.667 (6)	113
O1*B*—H1*BA*···O2*B*	0.82	2.15	2.618 (3)	116
O4*B*—H4*BA*···O2*A*	0.82	2.03	2.831 (3)	166
O4*B*—H4*BA*···O3*B*	0.82	2.42	2.696 (3)	100
O1*C*—H1*CA*···O2*C*	0.82	2.31	2.710 (3)	110
O1*C*—H1*CA*···O4*C*^{ii}	0.82	2.25	3.067 (4)	175
O4*C*—H4*CA*···O3*C*	0.86 (7)	2.37 (6)	2.650 (4)	100 (5)
O4*C*—H4*CA*···O2*C*^{iii}	0.86 (7)	2.01 (6)	2.749 (4)	143 (6)
C12*B*—H12*D*···O3*B*	0.97	2.44	2.811 (3)	103
C12*C*—H12*F*···O3*C*	0.97	2.45	2.799 (4)	102
C17*B*—H17*D*···O2*A*^{iv}	0.96	2.52	3.460 (4)	165
C18*A*—H18*A*···O1*A*	0.96	2.33	2.789 (4)	109
C18*B*—H18*D*···O1*B*	0.96	2.38	2.839 (4)	109
C18*C*—H18*G*···O1*C*	0.96	2.34	2.800 (5)	109
C4*C*—H4*CB*···O1*A*	0.97	2.54	3.510 (3)	176

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

The hydroxyl H atom attached at O4*C* was located in a difference map and isotropically refined. 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 0.96–0.98 Å. The U_{iso} values were set equal to $1.5U_{eq}$ of the carrier atom for hydroxyl and methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. Friedel pairs were merged before the final refinement as there is no significant anomalous dispersion for the determination of the absolute configuration.

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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