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7-Oxogedunin

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.047
wR factor = 0.110
Data-to-parameter ratio = 7.9

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

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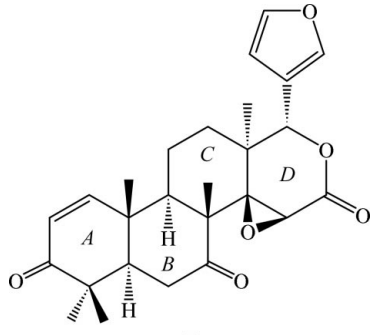
The title compound, 1-(3-furyl)-4b,7,7,10a,12a-pentamethyl-1,6a,7,10a,10b,11,12,12a-octahydronaphtho[2,1-*f*]oxireno[2,3-*d*]isochromene-3,5,8(3a*H*,4b*H*,6*H*)-trione, $C_{26}H_{30}O_6$, has been isolated from *Xylocarpus granatum* Koenig. All four rings are *trans*-fused. The four six-membered rings adopt distorted sofa, chair, twist-boat and half-chair conformations. The furan substituent is planar and attached equatorially to the lactone ring of the gedunin skeleton. The crystal structure is stabilized by C—H···O intramolecular and intermolecular interactions.

Comment

Xylocarpus granatum Koenig is a mangrove plant from the family Meliaceae, growing around the littoral of the tropical Indian ocean and extending to the Pacific islands. Limonoids from this plant have been reported to exhibit important biological activities such as antimalarial (MacKinnon *et al.*, 1997) and insect antifeedant (Champagne *et al.*, 1992), whereas the aqueous extract showed significant antifilarial activity (Zaridah *et al.*, 2001). The title compound, 7-oxogedunin, (I), was previously isolated from various genera of the Meliaceae family, such as *Guarea Guidona* (Lukacova *et al.*, 1982), *Xylocarpus moluccenis* (Mulholland & Taylor, 1992) and *Cedrela Odorata* (Paula *et al.*, 1997). It was found not to possess antitubercular, antimalarial or antitumor activities.



(I)

The title compound was isolated from the seeds of *Xylocarpus granatum* Koenig, which were collected from Nakhon-Si-Thammarat province in southern Thailand. As part of our research on bioactive constituents from Thai mangrove plants (Chantrapromma *et al.*, 2003; Fun *et al.*, 2003; Cheenpracha *et al.*, 2004), we report here the crystal structure of (I). The results will be useful for molecular modeling and biotransformation studies, which are the current interests of our research group. Although the title compound has no activity, a modification of its molecular structure to induce antimalarial activity and cytotoxicity will be under investigation.

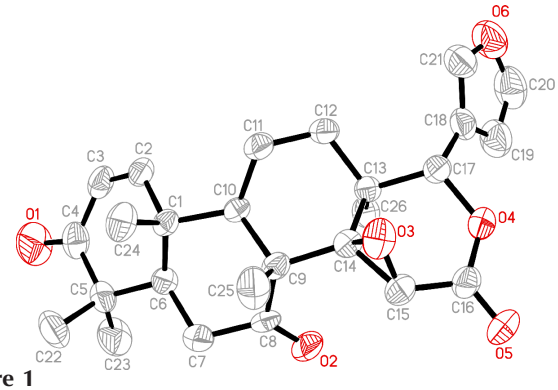


Figure 1
The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

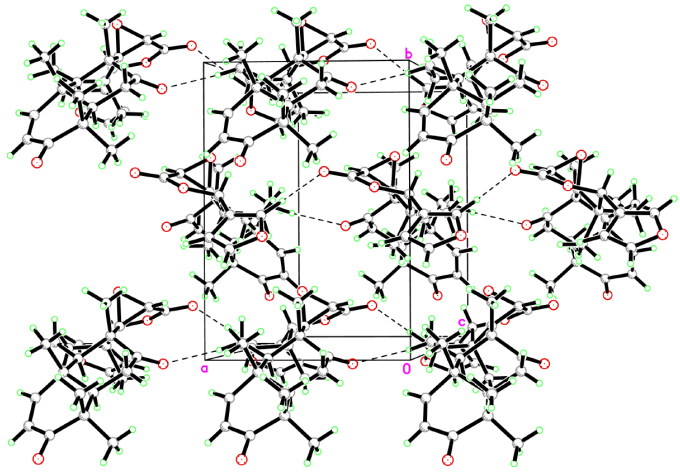


Figure 2
The packing in the crystal structure of (I), viewed approximately down the *c* axis. Dashed lines indicate hydrogen bonds.

The structure of (I) contains a four-ring *A*/*B*/*C*/*D* fused triterpenoid system, known as the gedunin skeleton (Fig. 1). The *A*/*B*, *B*/*C* and *C*/*D* ring junctions are *trans*-fused. The bond lengths and angles in (I) show normal values (Allen *et al.*, 1987) and are comparable with the corresponding values in the related compounds mexicanolide (Sanni *et al.*, 1987) and 6-hydroxymexicanolide (Chantrapromma *et al.*, 2004). The crystal structure of gedunin itself has been reported earlier (Toscano *et al.*, 1996).

The conformations adopted by rings *A*, *B*, *C* and *D* are distorted sofa, approximate chair, twist-boat and half-chair, respectively (Cremer & Pople, 1975). The distorted sofa conformation is induced by the α,β-unsaturated ketone function of ring *A*. The methyl groups are attached axially to rings *A*, *B* and *C* at atoms C1, C5, C9 and C13. The furan ring is planar to within 0.007 (4) Å and is attached equatorially to lactone ring D, the torsion angle C13—C17—C18—C21 being 89.2 (4)°. The conformations of the carbonyl groups C4═O1, C8═O2 and C16═O5 are: (−)-synclinal with O1—C4—C5—C22 = −63.7 (5)°, (−)-anticlinal with O2—C8—C9—C25 = −116.2 (4)° and (+)-anticlinal with O3—C15—C16—O5 = 137.5 (4)°.

The crystal structure is stabilized by C—H···O intra- and intermolecular interactions (Table 2). The latter link molecules together, forming a molecular chain along the *a* axis (Fig. 2).

Experimental

Air-dried seeds of *Xylocarpus granatum* Koeing (5.2 kg) were extracted with hexane, CH_2Cl_2 and MeOH, successively. The dichloromethane extract was dried under reduced pressure to yield a crude extract (68.67 g). The crude extract (26.14 g) was separated by fast column chromatography on silica gel and eluted initially with hexane enriched with ethyl acetate, followed by an increasing amount of methanol in ethyl acetate and finally with methanol. Each fraction was monitored by thin-layer chromatography; fractions that appeared similar were combined to yield 20 major fractions, denoted F1–F20. Fraction F5 (0.41 g) was further purified by crystallization to give (I) (0.25 g). Compound (I) was recrystallized from hexane–CH_2Cl_2–EtOAc (4:4:2) to give colorless single crystals after a few days (m.p. 533–535 K).

Crystal data

$C_{26}H_{30}O_6$	D_x = 1.301 Mg m^{-3}
M_r = 438.50	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 3780 reflections
a = 7.6623 (12) Å	θ = 1.6–28.3°
b = 11.1298 (18) Å	μ = 0.09 mm^{-1}
c = 13.350 (2) Å	T = 293 (2) K
β = 100.547 (3)°	Plate, colorless
V = 1119.2 (3) Å^3	0.25 × 0.19 × 0.08 mm
Z = 2	

Data collection

Siemens SMART CCD area detector diffractometer	2330 independent reflections
ω scans	2052 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	R_{int} = 0.031
T_{min} = 0.977, T_{max} = 0.993	θ_{max} = 26.0°
8802 measured reflections	$h = -9 \rightarrow 9$
	$k = -13 \rightarrow 13$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2 + 0.1209P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.110	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
S = 1.11	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
2330 reflections	
294 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.210 (4)	O6—C20	1.349 (6)
O2—C8	1.211 (4)	O6—C21	1.357 (5)
O5—C16	1.195 (4)	C2—C3	1.318 (5)
O3—C15	1.431 (4)	C18—C21	1.341 (5)
O3—C14	1.453 (4)	C18—C19	1.422 (5)
O4—C16	1.340 (4)	C19—C20	1.330 (6)
O4—C17	1.464 (4)		
C15—O3—C14	61.4 (2)	C23—C5—C22	108.1 (3)
C16—O4—C17	121.1 (2)		
O1—C4—C5—C22	−63.7 (5)	O3—C15—C16—O5	137.5 (4)
O1—C4—C5—C6	171.5 (4)	C14—C15—C16—O5	−153.4 (3)
O2—C8—C9—C25	−116.2 (4)	C13—C17—C18—C21	89.2 (4)
O2—C8—C9—C10	120.4 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11B···O2^{i}	0.97	2.35	3.288 (4)	162
C21—H21A···O5^{i}	0.93	2.43	3.361 (5)	176
C23—H23A···O1	0.96	2.50	2.846 (6)	101

Symmetry code: (i) $x-1, y, z$.

H atoms were placed in calculated positions, with 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 atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. In the absence of significant anomalous dispersion effects, Friedel pairs were merged before the final refinement. Owing to a large fraction of weak data at higher angles, the 2θ maximum was limited to 52°.

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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