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Key indicators

Single-crystal X-ray study
 $T = 297\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.042
 wR factor = 0.120
 Data-to-parameter ratio = 9.0

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

seco-Dukunolide F: a new tetranortriterpenoid from *Lansium domesticum* Corr.

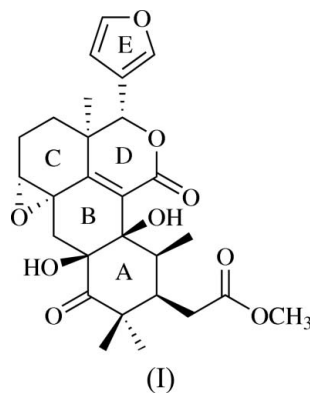
The title compound, methyl 2-[4-(3-furyl)-6b,10a-dihydroxy-3a,7,9,9-tetramethyl-6,10-dioxo-2,3,3a,6b,7,8,9,10,10a,11-decahydro-1a*H*,4*H*,6*H*-benzo[*h*][1]benzoxireno[3,2,1a-*de*]isochromen-8-yl]acetate, $\text{C}_{27}\text{H}_{32}\text{O}_9$, is a new tetranortriterpenoid which was isolated for the first time from the seeds of *Lansium domesticum* Corr. The four six-membered rings adopt chair, boat, envelope and screw-boat conformations. The furan substituent is planar and is attached equatorially to the lactone ring. $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into chains along [100] and adjacent chains are interconnected by $\text{C}-\text{H}\cdots\text{O}$ interactions.

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Comment

The title compound, (I), is a naturally occurring tetranortriterpene which was isolated for the first time from the seeds of *Lansium domesticum* Corr., a plant belonging to the Meliaceae family. *L. domesticum* Corr. has at least four cultivated forms or varieties, namely, langsat, duku, dukulangsat and longkong. This plant is widely cultivated for its edible fruit. In North Borneo, the bark and seeds have been used to treat malaria and fever (Burkill, 1966; Chai *et al.*, 1989). It has also been reported that the Ibans in Sarawak, Malaysia, used the langsat leaves to treat fever (Leaman *et al.*, 1995). As part of our ongoing studies of bioactive compounds from Thai medicinal plants (Chantrapromma *et al.*, 2005; Chantrapromma, Boonnak *et al.*, 2006; Chantrapromma, Fun *et al.*, 2006; Fun *et al.*, 2006), we have undertaken the X-ray crystal structure analysis of (I) in order to establish its molecular structure and relative stereochemistry. Compound (I) was not found to possess antimalarial, antitubercular or anti-tumour activities.



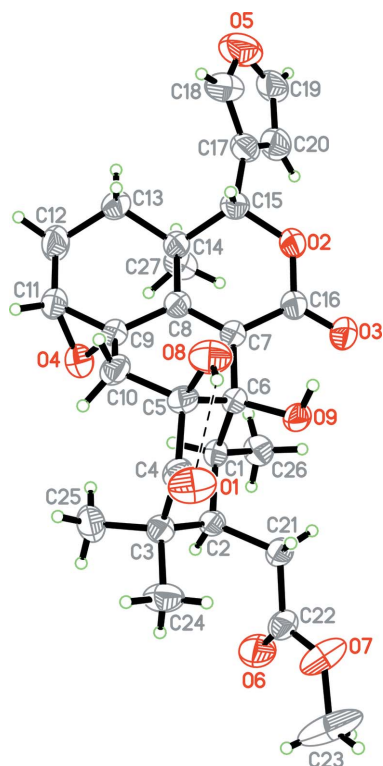


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. The O—H···O hydrogen bond is shown as a dashed line.

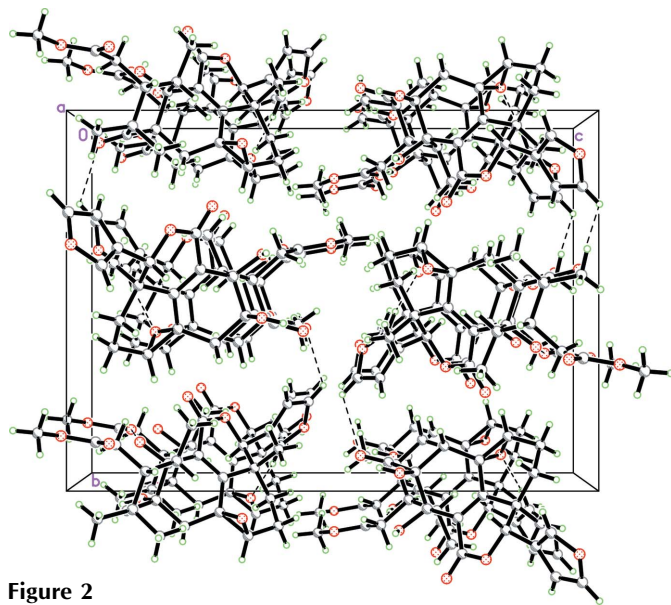


Figure 2
The crystal packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

A/B ring junction is *cis*-fused. The bond lengths and angles in (I) show normal values (Allen *et al.*, 1987) and are comparable to the corresponding values in a closely related compound, dukunolide F (Nishizawa *et al.*, 1988). The conformations adopted by rings *A*, *B*, *C* and *D* are chair, boat, envelope with atom C13 in the flap position [$Q = 0.488$ (3) Å, $\theta = 58.4$ (4)°

and $\varphi = 244.1$ (4)°] and screw-boat [$Q = 0.518$ (2) Å, $\theta = 66.2$ (2)° and $\varphi = 89.7$ (3)°; Cremer & Pople, 1975], respectively. The methyl group at C1 is equatorially attached to ring *A*. The conformation of the methyl acetate side chain (C21–C23/O6/O7) is (–)-antiperiplanar with a C1–C2–C21–C22 torsion angle of -147.41 (19)°. This side chain and two hydroxyl groups are in *cis* orientations (Fig. 1). The furan ring is planar within ± 0.005 (2) Å and is attached equatorially to the lactone ring *D*, the torsion angle C14–C15–C17–C20 being -86.0 (3)°. The methyl group attached at C14 is *cis* with respect to the furan ring and *trans* with respect to the two hydroxyl groups. The two hydroxyl groups have β -orientations, similar to those of dukunolide F (Nishizawa *et al.*, 1988) but in contrast with those of dukunolide A (Nishizawa *et al.*, 1985). One of the hydroxyl groups forms an intramolecular hydrogen bond, O8–H8···O1 (Fig. 1), with carbonyl atom O1. In addition, the molecular structure is stabilized by intramolecular C–H···O weak interactions (Table 1).

O–H···O intermolecular hydrogen bonds (Table 1) link the molecules into chains along [100]. Adjacent chains are interlinked *via* C–H···O hydrogen bonds (Fig. 2).

Experimental

The seeds (1.6 kg) of *L. domesticum* Corr., which were collected from Nakhon Si Thammarat province in the southern part of Thailand, were air-dried, powdered and then extracted twice with CH₂Cl₂ at room temperature (five days for each extraction) to yield a yellow-green viscous liquid (33.2 g). The crude extract was separated by flash column chromatography over silica gel and then eluted with hexane and three further solvents in order of increasing polarity, CH₂Cl₂, EtOAc and CH₃OH, to give 13 fractions (F1–F13). Fraction F5 (9.0 g) was washed with EtOAc followed by recrystallization from EtOAc–CH₂Cl₂ (1:1 *v/v*), affording colourless single crystals of compound (I) (2.0 g, m.p. 508–510 K).

Crystal data

C₂₇H₃₂O₉
 $M_r = 500.53$
 Orthorhombic, $P2_12_12_1$
 $a = 7.9634$ (5) Å
 $b = 14.6700$ (9) Å
 $c = 20.5420$ (13) Å
 $V = 2399.8$ (3) Å³

$Z = 4$
 $D_x = 1.385$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 297$ (2) K
 Block, colourless
 $0.50 \times 0.39 \times 0.34$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.952$, $T_{\max} = 0.965$

14158 measured reflections
 2973 independent reflections
 2814 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$
 $\theta_{\max} = 27.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.120$
 $S = 1.05$
 2973 reflections
 332 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0826P)^2 + 0.3239P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.30$ e Å⁻³
 $\Delta\rho_{\min} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O8—H8···O1	0.82	2.07	2.575 (3)	119
O9—H9···O6 ⁱ	0.82	2.09	2.910 (2)	174
C2—H2···O6	0.98	2.47	2.926 (3)	108
C18—H18···O4 ⁱ	0.93	2.58	3.452 (3)	157
C19—H19···O1 ⁱⁱ	0.93	2.55	3.096 (4)	118
C27—H27A···O2	0.96	2.60	2.977 (3)	104

Symmetry codes: (i) $x - 1, y, z$; (ii) $-x, y + \frac{1}{2}, -z + \frac{3}{2}$.

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 $U_{iso}(H)$ 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 hydroxyl and methyl groups. In the absence of significant anomalous dispersion effects, Friedel pairs were merged before the final refinement.

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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