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2-(9-Hydroxy-3a,5a,5b,8,8,11a-hexamethylicosahydro-1*H*-cyclopenta[*a*]chrysen-1-yl)propanoic acid (3*β*-hydroxylupan-29-oic acid)

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Key indicators

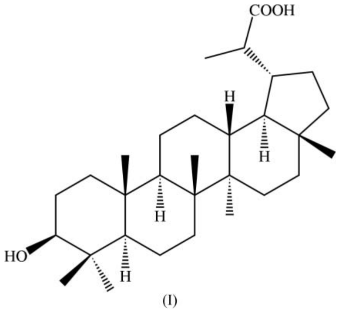
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.053
wR factor = 0.117
Data-to-parameter ratio = 8.4

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

The title compound, $C_{30}H_{50}O_3$, a lupane triterpene, was isolated from the leaves of *Ceriops decandra* (Griff.) Ding Hou. There are two crystallographically independent molecules in the asymmetric unit. In both molecules, the cyclopentane ring adopts an envelope conformation. In the crystal structure, molecules are interconnected into a two-dimensional network by intermolecular O—H···O hydrogen bonds.

Comment

Ceriops decandra (Griff.) Ding Hou (Rhizophoraceae) is a mangrove plant widely distributed from East Africa and Madagascar throughout tropical Asia and Queensland to Melanesia and Micronesia (Tomlinson, 1986). *C. decandra* has many local Thai names, *e.g.* Prong Khao and Prong Nu, and also a synonym of *Ceriops roxburghiana* Arn (Smitinand & Larsen, 1970). The bark of this plant has been used as a folk medicine for the treatment of diarrhoea, vomiting, amoebiasis and ulcers (Bamroongrugsa, 1999). An ethanol extract of the leaves has shown antinociceptive activity (Uddin *et al.*, 2005). The title compound, 3*β*-hydroxylupan-29-oic acid, (I), was previously isolated from *Gymnosporia wallichiana* (Kulshreshtha, 1977). As part of our research on bioactive compounds from Thai medicinal plants (Chantrapromma *et al.*, 2003, 2004; Waratchareeyakul *et al.*, 2004; Boonnak *et al.*, 2005), compound (I) was isolated for the first time from *Ceriops decandra* (Griff.) Ding Hou, collected from Phang-Nga province in the southern part of Thailand. We have undertaken the X-ray crystal structure analysis of (I) in order to establish its molecular structure and relative stereochemistry.



(I)

The asymmetric unit of (I) contains two crystallographically independent molecules, *A* and *B*, which have similar chiralities, bond lengths and angles (Fig. 1). The molecules are approximately related by a local twofold rotation axis. The bond lengths in (I) show normal values (Allen *et al.*, 1987). All the ring junctions in the lupane nucleus are *trans*-fused. In both molecules, the cyclohexane rings adopt chair conformations and the cyclopentane ring has an envelope conformation, with atom C17 displaced from the C18/C22/C23/C24 plane by −0.692 (7) Å in molecule *A* and 0.674 (7) Å in molecule *B*. The hydroxyl group is equatorially attached at atom C3. The C23—C22—C25—C27 torsion angle of −55.1 (5)° [−45.7 (5)° in *B*] describes the orientation of the propanoic acid group with respect to the lupane nucleus. The C18—C22—C25—C26 torsion angle is −170.1 (4)° [−161.8 (3)° in *B*].

The molecular structure is stabilized by C—H⋯O hydrogen bonds (Table 2). O—H⋯O intermolecular hydrogen bonds link the molecules into a two-dimensional network parallel to the *ac* plane (Fig. 2).

Experimental

Air-dried leaves of *Ceriops decandra* (Griff.) Ding Hou (3.9 kg) were ground and extracted with hexane (2 × 20 l) at room temperature. The mixture was filtered and concentrated under reduced pressure to give a crude hexane extract (66.4 g). The crude extract was separated by quick column chromatography (QCC) on silica gel and eluted initially with hexane, followed by ethyl acetate and finally with methanol to give nine fractions (B1–B9). Fraction B7 (2.7 g) was subjected to column chromatography with MeOH–CH_2Cl_2 (0.2:9.8 *v*/*v*) and further purified by recrystallization from $CHCl_3$–EtOAc–MeOH (4:4:1) for a few days to obtain colourless single crystals of compound (I) [m.p. 518–520 K, $[\alpha]_D^{28}$ −42.6° (*c* = 0.125, MeOH)].

Crystal data

$C_{30}H_{50}O_3$
$M_r = 458.70$
Monoclinic, $P2_1$
$a = 8.1669$ (8) Å
$b = 24.110$ (2) Å
$c = 14.4666$ (14) Å
$\beta = 104.057$ (2)°
$V = 2763.2$ (4) Å^3
$Z = 4$
$D_x = 1.103$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14 091 reflections
$\theta = 1.5$–25.0°
$\mu = 0.07$ mm^{-1}
$T = 293$ (2) K
Plate, colourless
0.23 × 0.15 × 0.08 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.988$, $T_{max} = 0.994$
14 091 measured reflections
4986 independent reflections
3296 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 25.0°$
$h = -9 \rightarrow 9$
$k = -23 \rightarrow 28$
$l = -14 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.117$
$S = 1.02$
4986 reflections
596 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.1104P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1*A*—C3*A*	1.452 (5)	O1*B*—C3*B*	1.445 (5)
O2*A*—C27*A*	1.234 (6)	O2*B*—C27*B*	1.236 (5)
O3*A*—C27*A*	1.264 (5)	O3*B*—C27*B*	1.284 (5)
O2*A*—C27*A*—O3*A*	122.9 (5)	O2*B*—C27*B*—O3*B*	123.8 (5)
O3*A*—C27*A*—C25*A*	118.1 (5)	O3*B*—C27*B*—C25*B*	116.0 (4)

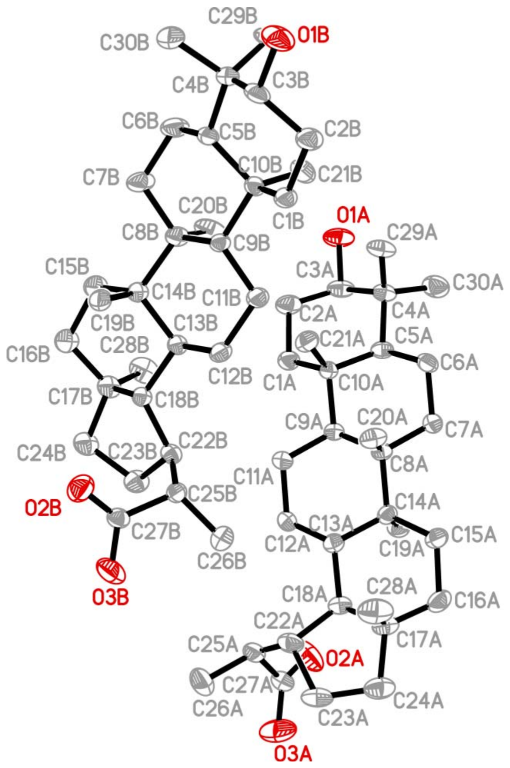


Figure 1
The asymmetric unit of (I), showing 30% probability displacement ellipsoids and the atomic numbering. For clarity, H atoms have been omitted.

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1*A*—H1*A*⋯O2*B*^{i}	0.82	1.91	2.705 (6)	161
O1*B*—H1*D*⋯O2*A*^{ii}	0.82	1.86	2.662 (6)	165
O3*A*—H3*A*⋯O1*A*^{iii}	0.82	1.98	2.724 (6)	151
O3*B*—H3*C*⋯O1*B*^{iv}	0.82	1.82	2.625 (5)	163
C18*A*—H18*A*⋯O2*A*^{v}	0.98	2.52	3.262 (6)	132
C18*B*—H18*B*⋯O2*B*^{v}	0.98	2.46	3.206 (5)	133
C23*A*—H23*B*⋯O3*A*^{v}	0.97	2.49	3.222 (7)	132
C23*B*—H23*D*⋯O3*B*^{v}	0.97	2.49	3.237 (6)	134
C30*A*—H30*A*⋯O1*A*^{v}	0.96	2.56	2.957 (7)	105
C30*B*—H30*D*⋯O1*B*^{v}	0.96	2.51	2.917 (7)	105

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z$; (ii) $-x, y-\frac{1}{2}, -z$; (iii) $x, y, z+1$; (iv) $x+1, y, z+1$; (v) x, 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 $1.5U_{eq}$ of the carrier atom for hydroxyl and 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 and the absolute configuration was assigned arbitrarily.

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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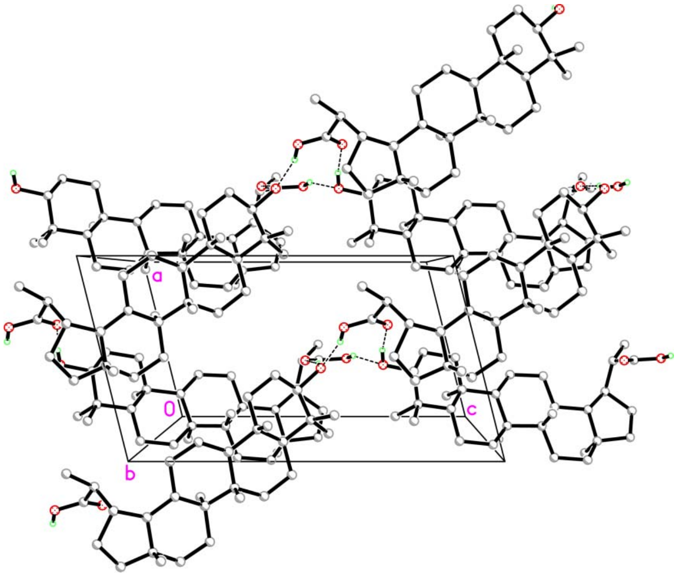

Figure 2
A view of the packing of (I). Only hydroxyl H atoms are shown. O—H···O hydrogen bonds are shown as dashed lines.

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bamroongrugsa, N. (1999). *Songklanakarin J. Sci. Technol.* **21**, 377–386.
Boonnak, N., Chantrapromma, S., Fun, H.-K., Anjum Ali, S., Rahman, A. & Karalai, C. (2005). *Acta Cryst.* E**61**, o410–o412.
Chantrapromma, S., Fun, H.-K., Ibrahim, A. R., Laphookhieo, S. & Karalai, C. (2003). *Acta Cryst.* E**59**, o1864–o1866.
Chantrapromma, K., Saewan, N., Fun, H.-K., Chantrapromma, S. & Rahman, A. A. (2004). *Acta Cryst.* E**60**, o312–o314.
Kulshreshtha, D. K. (1977). *Phytochemistry*, **16**, 1783–1785.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Smitinand, T. & Larsen, K. (1970). *Flora of Thailand*, Vol. 2, Part 1, pp. 10–12. Bangkok: The ASRCT Press.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tomlinson, P. B. (1986). *The Botany of Mangroves*, pp. 352–357. Cambridge University Press.
Uddin, S. J., Shilpi, J. A., Barua, J. & Rouf, R. (2005). *Fitoterapia*, **76**, 261–263.
Waratchareeyakul, W., Chantrapromma, S., Fun, H.-K., Razak, I. A., Karalai, C. & Ponglimanont C. (2004). *Acta Cryst.* E**60**, o1964–o1966.

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