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Lup-1,20(29)-dien-3-one, a triterpenoid from *Agyneia bacciformis* A. Juss

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.061
wR factor = 0.152
Data-to-parameter ratio = 12.0

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

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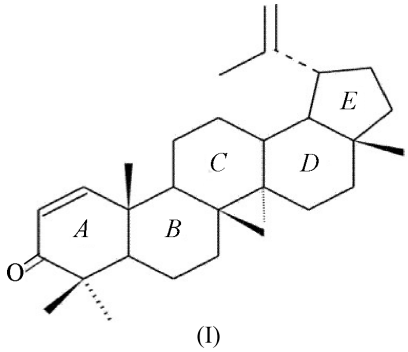
The crystal structure of glochidone [or lup-1,20(29)-dien-3-one], $C_{30}H_{46}O$, from the plant *Agyneia bacciformis* A. Juss has been determined. This natural product has anti-inflammatory activity. The rings *A*/*B*, *B*/*C*, *C*/*D* and *D*/*E* are *trans* fused. The molecules in the crystal structure are stabilized by C—H···O short contacts and van der Waals forces.

Comment

The title compound, (I), is an α,β-unsaturated ketone (Fig. 1). The bond distances and angles are comparable to those observed in a similar triterpene structure, that of alusenone (Ohki *et al.*, 1981) and lup-20(29)-en-3-one (Dampawan *et al.*, 1977). The C2—C3 and C19—C20 bonds assume partial double-bond character due to the adjacent double bond. The apparent shortening of the bond C30—C20 may be due to the high thermal vibration of atom C30. The lengthening of the bonds C8—C9 and C8—C14 in glochidone may be due to steric crowding at atoms C8 and C14, as observed in the structure of the triterpenoid lantadene A (Pattabhi & Sukumar, 1991). Methyl groups C23 and C27 are α-substituted and groups C24, C25, C26 and C28 are β-substituted (Fig. 2), which is in agreement with the chemical structure assignments. The rings *A*/*B*, *B*/*C*, *C*/*D* and *D*/*E* are *trans* fused. The molecular structure shows that the rings *A* and *E* are in distorted envelope conformations while rings *B*, *C* and *D* are in chair conformations (Cremer & Pople, 1975). A short H···O contact of 2.71 Å is observed between atoms H26*B* and O31($\frac{3}{2}-x$, $2-y$, $z-\frac{1}{2}$).



(I)

Experimental

Glochidone was extracted from the plant *Agyneia bacciformis* A. Juss, as reported by Purushothaman & Saradha (1982). Single crystals

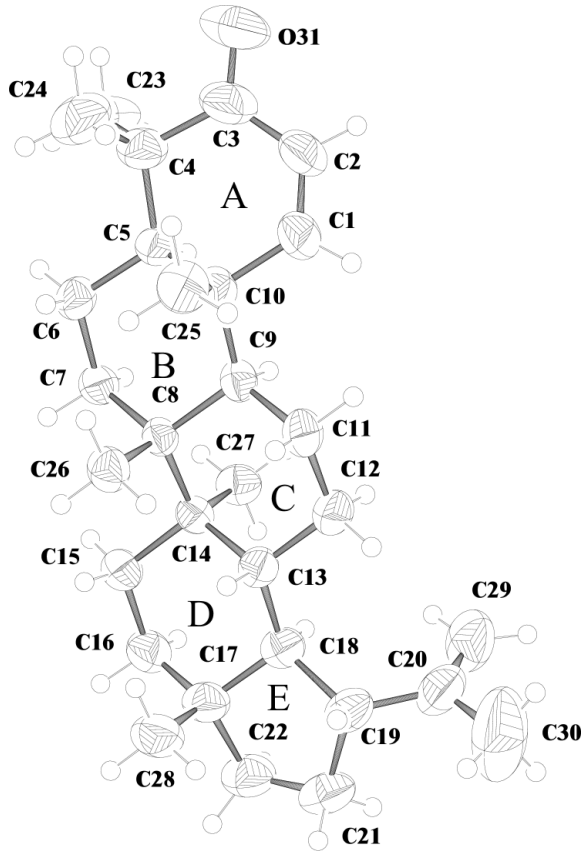


Figure 1
The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

of the compound were obtained from dimethyl fluoride solution at room temperature (293 K).

Crystal data

$C_{30}H_{46}O$	Mo $K\alpha$ radiation
M_r = 422.67	Cell parameters from 6028 reflections
Orthorhombic, $P2_12_12_1$	θ = 1.9–28.0°
a = 10.422 (4) Å	μ = 0.06 mm^{-1}
b = 13.881 (5) Å	T = 293 (2) K
c = 17.591 (6) Å	Block, colourless
V = 2544.8 (15) Å^3	0.48 × 0.40 × 0.40 mm
Z = 4	
D_x = 1.103 Mg m^{-3}	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2726 reflections with $I > 2\sigma(I)$
	R_{int} = 0.044
ω scans	θ_{max} = 28.0°
Absorption correction: none	$h = -13 \rightarrow 13$
22282 measured reflections	$k = -18 \rightarrow 18$
3423 independent reflections	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.079P)^2 + 0.3676P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.061	
$wR(F^2)$ = 0.152	
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
3423 reflections	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
286 parameters	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
H-atom parameters constrained	

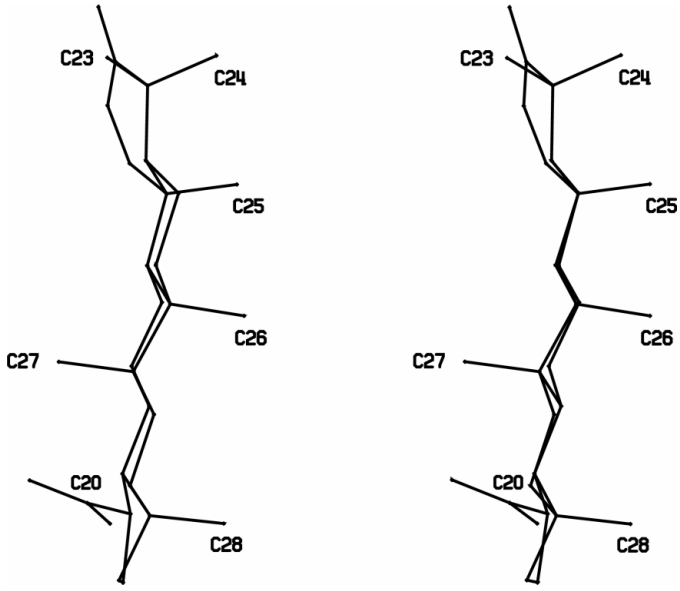


Figure 2
Stereoview of glochidone, showing the α-substituted (C23 and C27) and β-substituted (C24, C25, C26 and C28) methyl groups.

Table 1
Selected bond distances (Å).

C1—C2	1.322 (5)	C8—C14	1.593 (3)
C1—C10	1.495 (4)	C19—C20	1.493 (5)
C2—C3	1.449 (5)	C20—C29	1.363 (6)
C8—C9	1.560 (3)	C20—C30	1.438 (6)

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms. In the absence of significant anomalous scattering, Friedel pairs were merged, and the absolute configuration was assigned arbitrarily.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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