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Dammaradienyl acetate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.009 Å
R factor = 0.056
wR factor = 0.192
Data-to-parameter ratio = 8.9

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

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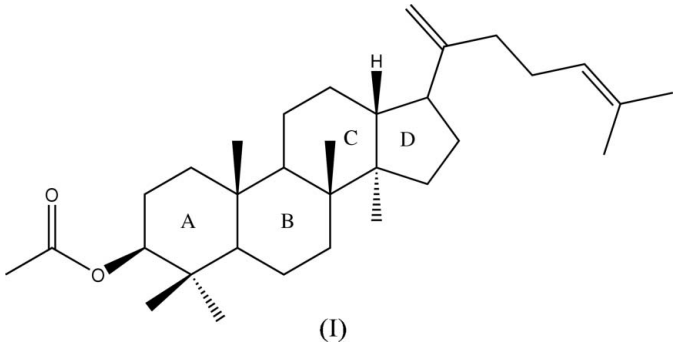
The title compound [systematic name: 4,4,8,10,14-pentamethyl-17-(6-methylhepta-1,5-dien-2-yl)hexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl acetate], $C_{32}H_{52}O_2$, contains a fused four-ring system. All three six-membered rings adopt chair conformations and the five-membered ring is twisted. The *A*/*B*, *B*/*C* and *C*/*D* ring junctions are all *trans*-fused. A chain running along the *b* axis is formed *via* C—H···O hydrogen bonds, and translation of the chain along the *a* and *c* axes generates the three-dimensional structure.

Comment

The title compound dammaradienyl acetate, (I), was originally isolated from the Indian plant *Commelina undulata*, which was shown to possess anticancer activity against lymphoid leukaemia in mice (PS 388) in the screening programme of the US National Institutes of Health (Sharma & Tandon, 1982). We have now isolated this compound from *Inula nervosa*. Here, the crystal structure of (I) is reported.



(I)

The skeleton of (I) is composed of a fused four-ring system, including three six-membered rings, *A* (C3–C7/C12), *B* (C7–C12) and *C* (C10–C16), and a five-membered ring, *D* (C15–C19). All the junctions are *trans*-fused, as indicated by their torsion angles (Table 1), which is similar to what is observed in 1-acetyl-24-*epi*-polacandrin (Simirgiotis *et al.*, 2003). All three six-membered rings, *A*, *B* and *C*, adopt chair conformations, as shown by their puckering parameters (Cremer & Pople, 1975) [q_2 = 0.039 (5), q_3 = 0.553 (5), Q = 0.554 (5) Å, θ = 4.4 (5) and φ = 70 (8)° for ring *A*; q_2 = 0.083 (5), q_3 = 0.564 (5), Q = 0.570 (5) Å, θ = 8.4 (5) and φ = 5(3)° for ring *B*; q_2 = 0.071 (5), q_3 = 0.585 (5), Q = 0.589 (5) Å, θ = 6.9 (5) and φ = 311 (4)° for ring *C*. The value for δ of 704.2° suggests that ring *D* is twisted about the C15—C16 bond [$\delta = 2P$ (Altona *et al.* 1968), P = 352.1 (4), $\tau(M)$ = 44.0 (3) for reference bond C15—C16, where P and $\tau(M)$ are the pseudorotation parameters (Rao *et al.*, 1981)]. The acetyloxyl group and the chain are equatorially

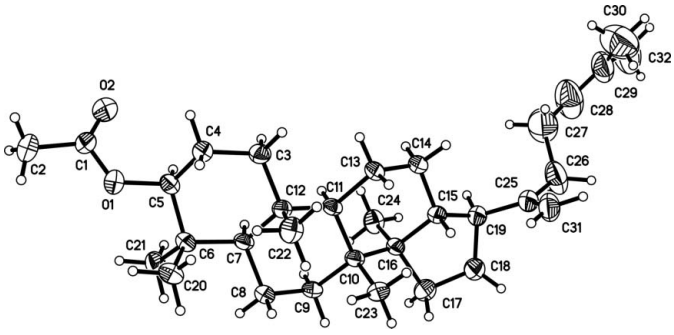


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

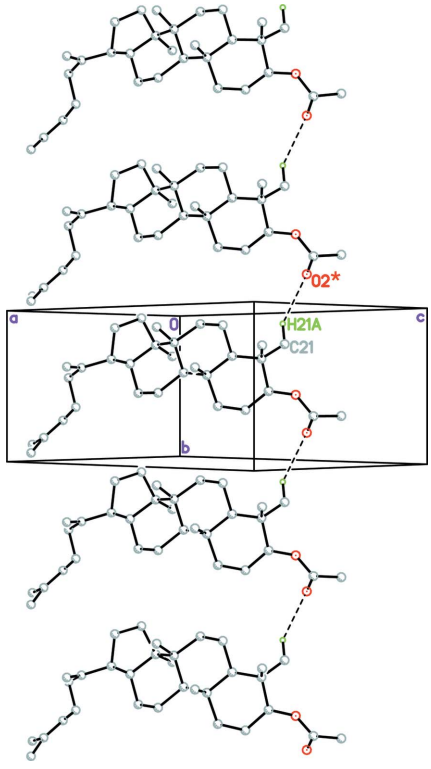


Figure 2
Part of the crystal structure of (I). Intermolecular C—H···O interactions are marked as dashed lines.

attached to rings *A* and *D*, respectively. The methyl groups C22, C23 and C24 are axially attached to rings *B* and *C*.

The hydrogen bond C21—H21*A*···O2^{i} links the molecules of (I) into a chain running along the *b* axis [C21···O2 = 3.432 (8) Å, H21*A*···O2 = 2.57 Å and C21—H21*A*···O2 = 149°; symmetry code: (i) *x*, *y* − 1, *z*; Fig. 2].

Experimental

The dried powdered herb *Inula nervosa* Wall. (14.8 kg) was extracted three times with hot ethanol (50 l × 3). The extract was evaporated under reduced pressure to yield a dark-green mass. This was treated with petroleum ether, chloroform and ethyl acetate. The petroleum ether fraction was chromatographed on a silica-gel column. The compound eluted with petroleum ether–ethyl acetate (10:1) crystallized from chloroform as needles (m.p. 422 K). Spectroscopic analysis: ^{1}H NMR (500 MHz, $CDCl_3$, δ, p.p.m.): 5.13 (1 H, *t sept*, *J* = 1.4 and 7.0 Hz, H24), 4.74 (1 H, *s*, H21), 4.70 (1 H, *s*, *J* = 1.5 Hz, H21), 4.49 (1 H, *dd*, *J* = 5.5 and 11.3 Hz, H3), 2.04, 1.63, 1.61 (each 3 H, *s*, –$COCH_3$, H26 and H27), 0.98, 0.88, 0.87,0.86, 0.85 (each 3 H, *s*), 0.83 (1 H, *m*, H5); ^{13}C NMR (125 MHz, $CDCl_3$, δ, p.p.m.): 170.9 (–$COCH_3$), 152.7 (C20), 131.3 (C25), 124.5 (C24), 107.5 (C21), 80.9 (C3), 55.9 (C5), 50.9 (C9), 49.4 (C14), 47.8 (C17), 45.3 (C13), 40.5 (C8), 38.8 (C22), 37.9 (C4), 37.1 (C10), 35.4 (C7), 34.2 (C1), 31.4 (C15), 28.9 (C23), 27.9 (C28), 27.1 (C2), 25.7 (C26), 24.9 (C16), 23.7 (C2), 21.4 (C11), 21.3 (–$COCH_3$), 18.2 (C6), 17.7 (C27), 16.5 (C30), 16.3 (C19), 15.9 (C18), 15.6 (C29). Crystals of (I) suitable for X-ray analysis were obtained from a chloroform solution by slow evaporation at room temperature.

Crystal data

$C_{32}H_{52}O_2$
M_r = 468.74
Monoclinic, $P2_1$
a = 11.660 (4) Å
b = 7.400 (3) Å
c = 16.632 (6) Å
β = 91.654 (6)°
V = 1434.5 (9) Å^3
Z = 2
D_x = 1.085 Mg m^{-3}
Mo *Kα* radiation
μ = 0.07 mm^{-1}
T = 298 (2) K
Prism, colourless
0.58 × 0.41 × 0.19 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.963, T_{max} = 0.988
7593 measured reflections
2735 independent reflections
1529 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.192
S = 1.00
2735 reflections
307 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1093P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected torsion angles (°).

C9—C10—C11—C13	−176.9 (3)	C8—C7—C12—C3	174.7 (4)
C23—C10—C11—C13	65.1 (5)	C6—C7—C12—C3	−51.5 (5)
C16—C10—C11—C13	−56.1 (4)	C8—C7—C12—C22	−65.8 (5)

The methyl H atoms were constrained to an ideal geometry, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C), but were allowed to rotate freely about the C—C bonds. All remaining H atoms were placed in geometrically idealized positions (C—H = 0.93–0.97 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C). In the absence of any significant anomalous scattering, Friedel pairs were merged during the final refinement and the absolute configuration is unknown.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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