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(3*S*,5*S*,10*S*,13*S*,14*S*,17*S*)-Methyl 3*β*-acetyl-25,26,27-trisnorlanost-8-en-24-oate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.005 Å
R factor = 0.064
wR factor = 0.173
Data-to-parameter ratio = 12.4

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

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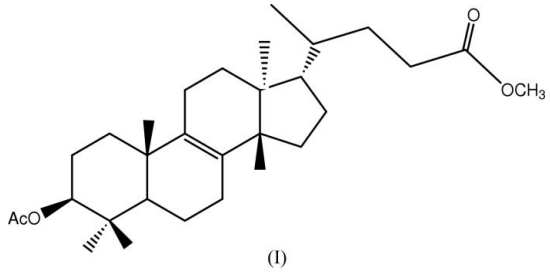
The absolute configuration of the title compound, $C_{30}H_{48}O_4$, was assigned by reference to a known chiral centre. The conformations of the four fused rings constituting the core of the molecule are comparable to those of some other triterpenes from the same family.

Comment

Carbonyl derivatives of triterpenes exhibit well known pharmacological activities (Akihisa *et al.*, 1996). In order to prepare similar derivatives with high added value, we have undertaken a scientific project based on the synthesis and characterization of such compounds (Benharref & Lavergne, 1985; Mazoir, Auhmani, Ait Itto *et al.*, 2004; Mazoir, Auhmani, Dakir *et al.*, 2004). The title compound, (I), was derived from eupho-lanosta-8,24-dien-3*β*-ol, a major triterpene isolated from *Euphorbia resinifera* latex. The structure of (I) was established by ^{1}H and ^{13}C NMR and confirmed by its single-crystal X-ray structure.



(I)

The core of the molecule consists of three six-membered and one five-membered fused rings (Fig. 1). A comparison of (I) with triterpenes already synthesized and characterized (Auhmani *et al.*, 2005; Daoubi *et al.*, 2001; Hosoe *et al.*, 2000) reveals that, despite the presence of different substituents on the five- and six-membered rings, the conformations of the rings are very similar; the five-membered ring in (I) adopts a twist conformation [q_2 = 0.466 (4) Å and φ_2 = 13.9 (5)°], while rings C8/C9/C11–C14 [θ = 128.6 (3)° and φ = 60.8 (5)°] and C5–C10 [θ = 49.9 (4)° and φ = 17.1 (5)°] adopt half-boat conformations and ring C1–C5/C10 adopts a chair conformation [θ = 3.2 (4)° and φ = 65.0 (7)°] (Cremer & Pople, 1975).

Experimental

Oxidation with $RuCl_3{\cdot}3H_2O$ of eupho-lanosta-8,24-dien-3*β*-ol, isolated from *Euphorbia resinifera* latex, followed by esterification then acetylation reactions, led to the title compound, (I), in 75% yield. Single crystals were obtained by evaporation of a methanol solution at 277 K. ^{1}H NMR ($CDCl_3$): δ 4.44 (H-3, *dd*, J_1 = 12 Hz, J_2 =

4 Hz), 3.59 (CH_3 of ester group), 2.09 (CH_3 of acetyl group), 2.25 (H-23, *m*), 0.76 (H-25, *s*), 0.79 (H-26, *s*), 0.95 (H-27, *s*); ^{13}C NMR ($CDCl_3$): δ 81.07 (C-3), 51.19 (C-23), 171.18 (CO of acetyl group), 174.87 (CO of ester group), 133.65 (C-8), 134.10 (C-9).

Crystal data

$C_{30}H_{48}O_4$	Mo $K\alpha$ radiation
M_r = 472.71	Cell parameters from 27772 reflections
Orthorhombic, $P2_12_12_1$	θ = 1.3–28.3°
a = 6.4779 (1) Å	μ = 0.07 mm^{-1}
b = 16.3089 (2) Å	T = 293 (2) K
c = 26.2448 (5) Å	Prism, colourless
V = 2772.78 (8) Å^3	0.4 × 0.2 × 0.2 mm
Z = 4	
D_x = 1.132 Mg m^{-3}	

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.069
φ and ω scans	θ_{max} = 28.3°
27772 measured reflections	$h = -8 \rightarrow 8$
3793 independent reflections	$k = -21 \rightarrow 21$
2873 reflections with $I > 2\sigma(I)$	$l = -34 \rightarrow 34$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0752P)^2 + 0.9128P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.064	
$wR(F^2)$ = 0.173	
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
3793 reflections	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
307 parameters	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
H-atom parameters constrained	

All H atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.96 Å and U_{iso}(H) = 1.2U_{eq}(C), except for the methyl groups, which were allowed to rotate freely about their C—C bond. Owing to the absence of any significant anomalous scatterers in the molecule, Friedel pairs were merged before the final refinement. The enantiomer has been assigned by reference to an unchanging chiral centre in the synthetic procedure.

Data collection: *KappaCCD Reference Manual* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

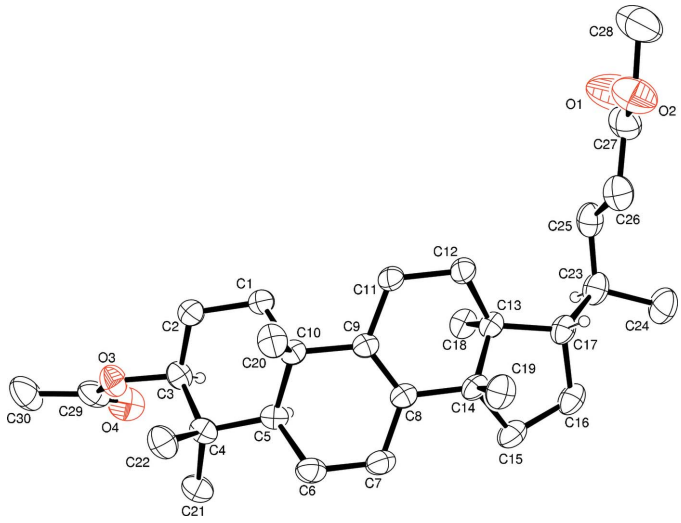


Figure 1
View of (I), showing the atom-labelling scheme and displacement ellipsoids drawn at the 30% probability level. Most of the H atoms have been omitted for clarity.

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