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(4S,5S,10S,13R,14R,17R)-8α,9α-Epoxy-4α,14α-dimethyl-5α-cholestan-3-one

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Key indicators

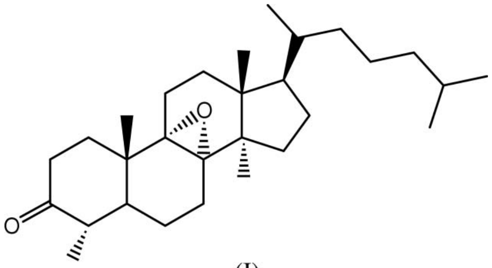
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.071
wR factor = 0.172
Data-to-parameter ratio = 12.6

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

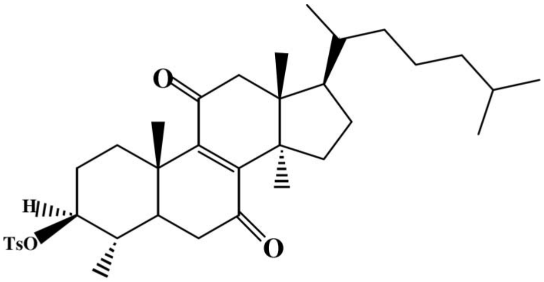
The stereochemistry of the oxirane bridge of the title compound, $C_{29}H_{48}O_2$, has been confirmed by single-crystal X-ray diffraction.

Comment

In our efforts to prepare compounds with potential pharmacological activities (Auhmani *et al.*, 2005; Mazoir, Liazid *et al.*, 2005), we undertook the synthesis of triterpenes functionalized with an oxirane bridge. It is well known that epoxidation of olefins affords a straightforward way to achieve such goals (Sharpless, 2002). Thus, the oxidation of 4α,14α-dimethyl-5α-cholest-8-en-3β-ol, (1), a major triterpene isolated from *Euphorbia officinarum* latex (Benharref & Lavergne, 1985; Mazoir, Giorgi & Auhman, 2005), with chromic anhydride (Auhmani *et al.*, 2005), followed by epoxidation using a stoichiometric quantity of *meta*-chloroperbenzoic acid (*m*-CPBA), gives the title compound, (I), as the only product. The structure of (I) was established by ^{1}H and ^{13}C NMR and confirmed by single-crystal X-ray diffraction.



(I)



(II)

The core of the molecule of (I) consists of one five-membered and three six-membered fused rings, with the oxirane bridge linking the two C atoms, C8 and C9, and *cis* to the methyl groups attached to atoms C4 and C14 (Fig. 1).

The crystal packing shows that molecules of (I) form extended sheets parallel to the *ac* plane (Fig. 2). The comparison of the molecular structure of (I) with that of a related triterpene bearing a double bond at C8=C9, (II) (Auhmani *et al.*, 2005), an analogue of the synthetic precursor of (I), reveals that the epoxidation of the olefin does not change the conformations of the rings within the molecule during the oxidation process: the r.m.s. deviation between (I) and (II), calculated with the 17 C atoms constituting the core of each molecule, is 0.2 Å. In conclusion, the stereochemistry of the four rings is conserved during the conversion of the olefin to the epoxide.

Experimental

The oxidation of 4α,14α-dimethyl-5α-cholest-8-en-3β-ol, (1), isolated from the latex of *Euphorbia officinarum*, was carried out using chromic anhydride in acetone at 273 K for 35 min. The product resulting from oxidation was dissolved in chloroform (50 ml), and 2 equivalents of *meta*-chloroperbenzoic acid (*m*-CPBA) were added. The mixture was stirred at room temperature for 3 h and yielded compound (I) in 85% yield. Suitable crystals were obtained by evaporation of a hexane solution at 277 K. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 0.73 (H18, *s*), 0.77 (H26, *d*, $J = 2$ Hz), 0.80 (H27, $J = 2$ Hz), 0.82 (H21, *d*, $J = 6$ Hz), 0.83 (H19, *s*), 0.87 (H29, *d*, $J = 6.2$ Hz); ^{13}C NMR (75 MHz, CDCl3, δ, p.p.m.): 36.3 (C1), 36.42 (C2), 212.40 (C3), 44.5 (C4), 49.2 (C5), 22.7 (C6), 22.00 (C7), 69.59 (C8), 68.26 (C9), 36.40 (C10), 19.40 (C11), 26.50 (C12), 36.32 (C20), 19.60 (C21), 36.20 (C22), 24.10 (C23), 39.50 (C24), 28.10 (C25), 21.50 (C26), 21.80 (C27), 16.50 (C28), 24.50 (C29).

Crystal data

$C_{29}H_{48}O_2$
$M_r = 428.67$
Monoclinic, $P2_1$
$a = 13.7023$ (4) Å
$b = 6.3493$ (2) Å
$c = 15.6206$ (5) Å
$\beta = 101.721$ (1)°
$V = 1330.60$ (7) Å^3
$Z = 2$
$D_x = 1.070$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11835 reflections
$\theta = 1.8$–28.4°
$\mu = 0.06$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.5 × 0.2 × 0.2 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: none
11835 measured reflections
3532 independent reflections
2892 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.07$
$\theta_{\rm max} = 28.4°$
$h = -18 \rightarrow 18$
$k = -8 \rightarrow 8$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.172$
$S = 1.10$
3532 reflections
280 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0662P)^2 + 0.4609P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.31$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.96 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$, except for the methyl groups, which were allowed to rotate freely about their C—C bond, with C—H distances constrained to 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged. The absolute configuration was assigned on the basis of the known configuration of the starting material.

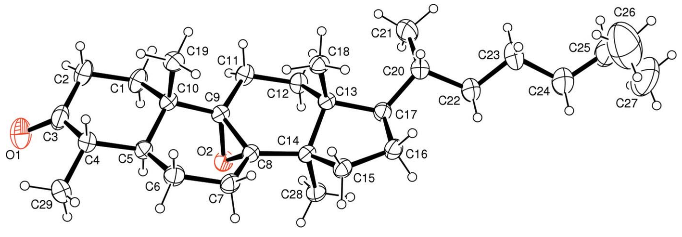


Figure 1
A view of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by circles of arbitrary size.

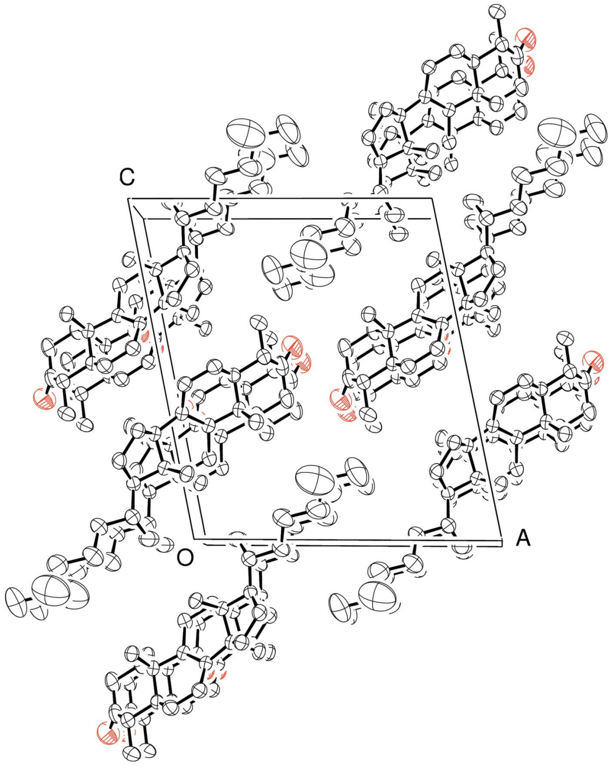


Figure 2
A view of the crystal packing of (I). H atoms have been omitted for clarity.

Data collection: *KappaCCD Server Software* (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: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

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