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(23*S*,25*R*)-3β,23-Dihydroxy-5-spirosten-3-yl acetate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.103
Data-to-parameter ratio = 9.5

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

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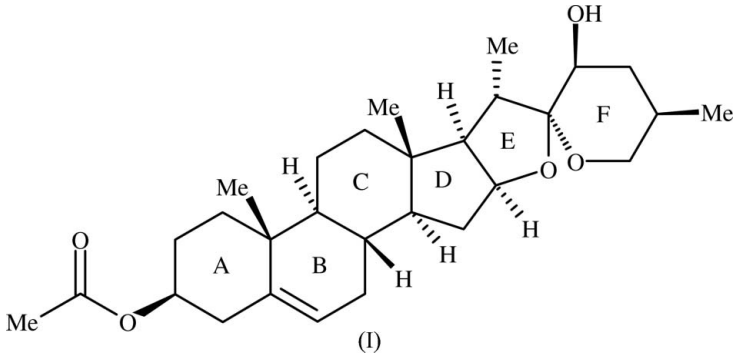
In the title compound, $C_{29}H_{44}O_5$, the ester substituent in ring *A* is equatorial. The six-membered rings *A* and *C* have chair conformations and ring *B* has a half-chair conformation. The *B*/*C* and *C*/*D* ring junctions are *trans*, whereas the tetrahydrofuran ring *E* is *cis*-fused to the cyclopentane ring *D*. An intermolecular O—H···O hydrogen bond is observed.

Comment

Steroid sapogenins are widespread in nature. Some of these compounds have served as starting materials in the synthesis of bioactive steroids that range from mammal, plant or insect hormones to steroid drugs. Some naturally occurring steroid sapogenins have been reported to exert antiproliferative activity in different lines of tumor cells (Raju *et al.*, 2004). As part of our studies on different bioactive steroids, the title compound, (I), is being extensively used as a starting material for the synthesis of steroids with potential activity as plant growth regulators or antiproliferative agents.



(I)

Fig. 1 shows the molecular structure of (I). The molecule contains a six-fused-ring system, the first four (*A*, *B*, *C* and *D*) of which are typical of the androstene steroid skeleton. The methyl groups at C10 and C13 are β in the steroid structure. The presence of the acetoxy group bonded to C3 does not disturb the chair conformation of ring *A* [puckering parameters (Cremer & Pople, 1975) Q = 0.558 (3) Å, θ = 9.3 (3)°, φ = 106 (2)°, if the calculation starts from C1 to C10 and proceeds in a clockwise direction]; all asymmetry parameters are less than 2.0 (3)° (Duax *et al.*, 1976). Rotational symmetry is dominant; ring *A* has a pseudo-C_2 axis which bisects the C3—C4 bond with asymmetry parameters ΔC_2(C3—C4) = 1.97 (4)° and ΔC_s(C3) = 0.09 (3)°. The average magnitude of the torsion angles is 54.2 (4)°. Ring *B* assumes an almost perfect half-chair conformation [Q = 0.453 (3) Å, θ = 49.6 (4)°, φ = 151 (5)°, if the calculation starts from C5 to C10 and

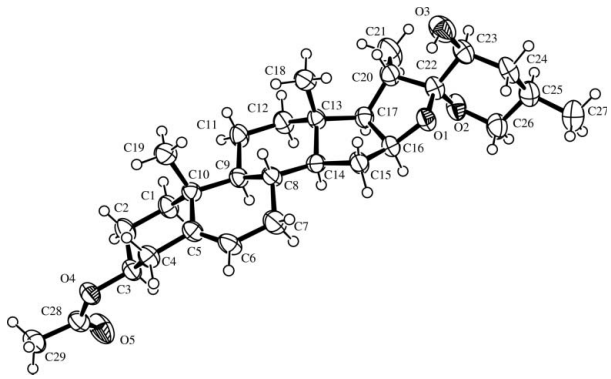


Figure 1
The structure of (I), with displacement ellipsoids at the 50% probability level.

proceeds in a clockwise direction]; the local twofold axis bisects the C5═C6 double bond with asymmetry parameter ΔC_2(C5═C6) = 0.82 (3)°. The *B*/*C* and *C*/*D* rings junctions are *trans*. The acetoxy group is β to the skeleton and arranged in such a way that the carbonyl O atom nearly eclipses the axial H atom at the 3-position of the ring. The tetrahydrofuran ring *E* is *cis*-fused to the cyclopentane ring *D*. The bond distances and bond angles are close to the expected values (Honda *et al.*, 1996). The molecules are linked *via* O—H···O hydrogen bonds (Table 2), forming a ribbon structure along the [$\bar{1}$10] direction.

Experimental

Treatment of the previously reported (25*R*)-3β-acetoxyspirost-5-en-23-one (1 g, 2.1 mmol) (Iglesias-Arteaga *et al.*, 2004) with sodium borohydride (0.8 g, 2.1 mmol) in methanol (5 ml) afforded compound (I) (yield 0.60 g, 60%). Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate solution.

Crystal data

$C_{29}H_{44}O_5$
M_r = 472.64
Orthorhombic, $P2_12_12_1$
a = 8.4510 (6) Å
b = 9.3980 (11) Å
c = 33.404 (3) Å
V = 2653.0 (4) Å^3
Z = 4
D_x = 1.183 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
Prism, colorless
0.60 × 0.27 × 0.27 mm

Data collection

Bruker *P*4 diffractometer
$2\theta/\omega$ scans
Absorption correction: none
3907 measured reflections
2989 independent reflections
2090 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 26°
3 standard reflections
every 97 reflections
intensity decay: 18.9%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.103
S = 1.06
2989 reflections
316 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0327P)^2 + 0.3392P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0088 (7)

Table 1
Selected geometric parameters (Å, °).

C3—O4	1.459 (3)	C22—O1	1.422 (4)
C5—C6	1.316 (4)	C26—O2	1.433 (4)
C16—O1	1.432 (3)	C28—O5	1.191 (4)
C22—O2	1.419 (4)	C28—O4	1.336 (4)
C10—C1—C2—C3	−56.6 (4)	C2—C1—C10—C5	48.2 (4)
C1—C2—C3—C4	60.7 (4)	C8—C9—C10—C5	−41.6 (3)
C2—C3—C4—C5	−59.1 (3)	C8—C9—C11—C12	52.0 (4)
C3—C4—C5—C6	−125.1 (3)	C9—C11—C12—C13	−53.2 (4)
C3—C4—C5—C10	53.8 (4)	C11—C12—C13—C14	53.6 (3)
C5—C6—C7—C8	14.7 (6)	C12—C13—C14—C8	−60.0 (4)
C6—C7—C8—C9	−41.8 (4)	C15—C16—C17—C20	125.4 (3)
C7—C8—C9—C10	56.9 (4)	C29—C28—O4—C3	−176.6 (3)
C4—C5—C10—C1	−47.0 (3)	C2—C3—O4—C28	85.0 (3)
C6—C5—C10—C9	13.3 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O5^{i}	0.90 (4)	2.08 (4)	2.909 (4)	153 (4)

Symmetry code: (i) $x-1, y+1, z$.

The H atom of the hydroxy group (O3/H3) was located in a difference map and refined isotropically with U_{iso}(H) = 1.5U_{eq}(O). H atoms attached to C atoms were placed in geometrically idealized positions and refined as riding on their parent atoms, with C—H = 0.93–0.98 Å and with U_{iso}(H) = 1.2U_{eq}(C), or 1.5 U_{eq}(C) for methyl groups. In the absence of significant anomalous scattering, Friedel pairs were merged. The absolute configuration of (I) was assumed to be the same as that of related compounds (Iglesias-Arteaga *et al.*, 2004), which is consistent with that previously predicted from the synthetic route.

Data collection: *XSCANS* (Siemens, 1993); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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References

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). *Topics in Stereochemistry*, Vol. 2, edited by E. L. Eliel & N. Allinger, pp. 271-283. New York: John Wiley.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Honda, T., Fujii, I., Hirayama, N., Ishikawa, D., Kawagishi, H., Song, K.-S. & Yoo, I.-D. (1996). *Acta Cryst.* C**52**, 1550–1552.
Iglesias-Arteaga, M. A., Sandoval-Ramírez, J., Mata-Esma, M. Y., Viñas-Bravo, O. & Bernès, S. (2004). *Tetrahedron Lett.* **45**, 4921–4926.
Raju, J., Patlolla, J. M. R., Swamy, M. V. & Rao, C. V. (2004). *Cancer Epidemiol. Biomarkers Prev.* **13**, 1392–1398.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1993). *XSCANS*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.