

Noureddine Mazoir,<sup>a</sup> Moha Berraho,<sup>b</sup> Bernard Fraïsse,<sup>c</sup> Ahmed Benharref<sup>a</sup> and Nouzha Bouhmaid<sup>d\*</sup>

<sup>a</sup>Laboratoire de Substances Naturelles, Faculté des Sciences Semlalia, BP 2390 Bd My Abdallah, 40000 Marrakech, Morocco,

<sup>b</sup>Laboratoire de Chimie de Coordination, Faculté des Sciences Semlalia, BP 2390 Bd My Abdallah, 40000 Marrakech, Morocco,

<sup>c</sup>Laboratoire Structures, Propriétés et Modélisation des Solides (SPMS), UMR CNRS 8580, Ecole Centrale Paris, Grande Voie des Vignes, 92295 Châtenay-Malabry, France, and

<sup>d</sup>Laboratoire Sciences des Matériaux (LSM), Faculté des Sciences Semlalia, BP 2390 Bd My Abdallah, 40000 Marrakech, Morocco

Correspondence e-mail: nouzha@ucam.ac.ma

#### Key indicators

Single-crystal X-ray study

$T = 100\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$

Disorder in main residue

$R$  factor = 0.059

$wR$  factor = 0.157

Data-to-parameter ratio = 20.5

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

## (3*S*,4*S*,5*S*,10*S*,13*R*,14*R*,17*R*)-4*α*,14*α*-Dimethyl-3*β*-tosyl-5*α*-ergost-8-ene-7,11,24-trione at 100 K

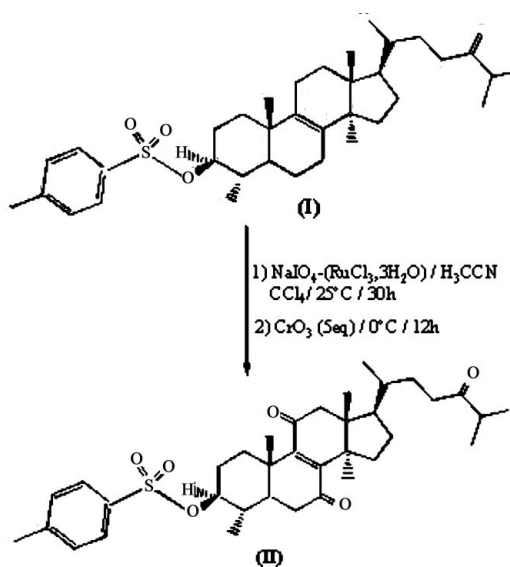
The title compound,  $\text{C}_{36}\text{H}_{50}\text{O}_6\text{S}$ , forms an extended sheet of four fused rings which exhibit different conformations, as already observed in previously reported triterpene structures. There are weak intra- and intermolecular  $\text{C}-\text{H}\cdots\text{O}$  interactions. A weak  $\text{C}-\text{H}\cdots\pi$  interaction also occurs, involving the tosyl group.

Received 24 February 2006

Accepted 26 April 2006

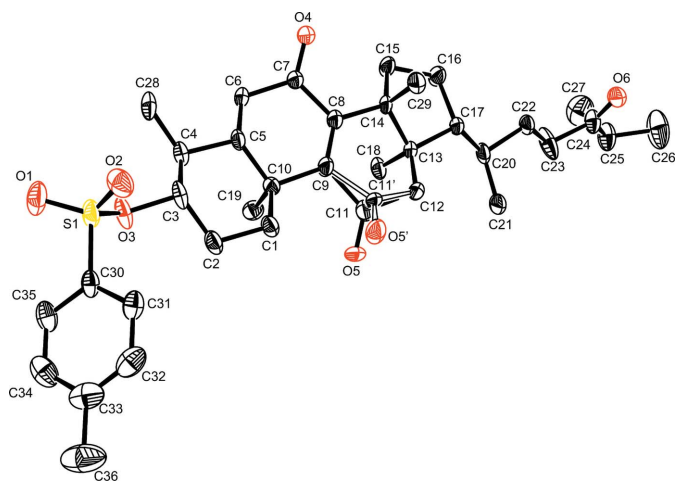
#### Comment

The oxidation of triterpenes resulting from *Euphorbia officinarum* latex gives carbonyl derivatives endowed with pharmacological activities (Akihisa *et al.*, 1996; Smith *et al.*, 2001). The title compound, (II), was obtained by oxidation of 3*β*-tosyl-5*α*-ergost-8,24-diene with ruthenium trichloride (Mazoir, Giorgi & Auhmani, 2005), followed by allylic oxydation using chromic anhydride (Auhmani *et al.*, 2005).

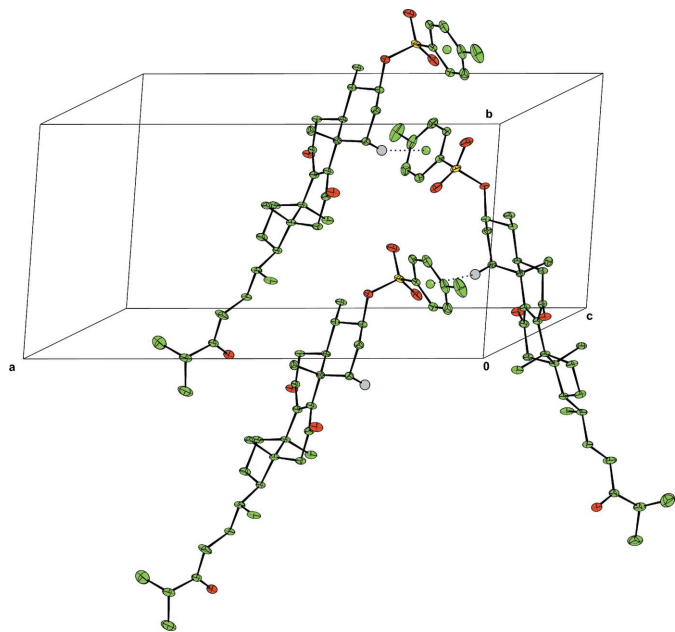


The molecule of (II) consists of an extended sheet containing one five-membered and three six-membered fused rings. Similar conformations were obtained for recently reported structures having the same molecular cores but with different substituents (Daoubi *et al.*, 2001; Auhmani *et al.*, 2005; Mazoir, Giorgi & Auhmani, 2005).

Calculation of the puckering parameters (Cremer & Pople, 1975) for the five-membered ring gives  $\theta_2 = 0.485$  (4)  $\text{\AA}$  and  $\varphi_2 = -163.2$  (5) $^\circ$ , corresponding to a twist conformation. The non-disordered six-membered rings adopt different conformations, namely a half-chair for the C5–C10 ring [ $\theta = 40.2$  (5) $^\circ$  and  $\varphi = 16.4$  (7) $^\circ$ ] and a chair conformation for the C1–C5/C10 ring [ $\theta = 3.0$  (4) $^\circ$ ]. The twist and chair conformations of the


**Figure 1**

The molecular structure of (II), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity. Both disorder components are shown.


**Figure 2**

The crystal packing of (II). C—H... $\pi$  interactions are indicated by dashed lines. H atoms not involved in the C—H... $\pi$  bonding have been omitted for clarity.

C13–C17 and C1–C5/C10 rings, respectively, were also observed in a previous study of (3*S*,4*S*,5*S*,10*S*,13*R*,14*R*,17*R*)-4 $\alpha$ ,14 $\alpha$ -dimethyl-3 $\beta$ -tosyl-5 $\alpha$ -cholest-8-ene-7,11-dione (Auhmani *et al.*, 2005). The S atom has a distorted tetrahedral environment, as already observed in a related compound (Auhmani *et al.*, 2005)

All the O atoms are involved in weak C—H...O inter- or intramolecular interactions (Table 1). An interesting feature is the occurrence of a weak C—H... $\pi$  interaction between atom H11 and the tosyl ring (Fig. 2); the corresponding structural descriptors (Nishio *et al.*, 1998) are given in Table 1. A similar interaction also occurs in (3*S*,4*S*,5*S*,10*S*,13*R*,14*R*,17*R*)-4 $\alpha$ ,14 $\alpha$ -

dimethyl-3 $\beta$ -tosyl-5 $\alpha$ -cholest-8-ene-7,11-dione (Auhmani *et al.*, 2005).

## Experimental

The oxidation of 3 $\beta$ -tosyl-5 $\alpha$ -ergosta-8,24-diene, a derivative triterpene from *Euphorbia officinarum* latex, was carried out using four equivalents of NaIO<sub>4</sub> with catalytic oxidation using RuCl<sub>3</sub>·3H<sub>2</sub>O in acetonitrile and carbon tetrachloride at 298 K for 30 h. The ketone obtained was dissolved in acetone (50 ml) and treated with five equivalents of chromic anhydride. The mixture was stirred for 12 h at 273 K to give (3*S*,4*S*,5*S*,10*S*,13*R*,14*R*,17*R*)-4 $\alpha$ ,14 $\alpha$ -dimethyl-3 $\beta$ -tosyl-5 $\alpha$ -ergost-8-ene-7,11,24-trione, (II), in 75% yield. Suitable crystals were obtained by evaporation of a methanol solution at 277 K. Spectroscopic analysis: <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>,  $\delta$ , p.p.m.): 4.08 (H3, *ddd*,  $J_1 = 11$  Hz,  $J_2 = 11$  Hz,  $J_3 = 3$  Hz), 7.78 (2H2', *d*,  $J = 8.8$  Hz), 7.35 (2H3', *d*,  $J = 8.8$  Hz), 2.48 (H3-5'), <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>,  $\delta$ , p.p.m.): 33.16 (C1), 26.08 (C2), 86.38 (C3), 35.80 (C4), 47.35 (C5), 38.28 (C6), 200.68 (C7), 151.44 (C8), 150.91 (C9), 40.98 (C10), 202.34 (C11), 51.50 (C12), 49.08 (C13), 47.55 (C14), 32.10 (C15), 27.30 (C16), 49.22 (C17), 16.90 (C18), 16.41 (C19), 35.86 (C20), 18.44 (C21), 38.19 (C22), 29.83 (C23), 214.54 (C24), 35.64 (C25), 21.72 (C26), 22.40 (C27), 14.5 (C28), 16.41 (C29), 144.72 (C1'), 134.62 (C4'), 129.86 (C2'), 127.80 (C3'), 18.44 (C5').

## Crystal data

C <sub>36</sub> H <sub>50</sub> O <sub>6</sub> S	Z = 4
$M_r = 610.82$	$D_x = 1.244$ Mg m <sup>-3</sup>
Monoclinic, C2	Mo K $\alpha$ radiation
$a = 22.8485$ (11) Å	$\mu = 0.14$ mm <sup>-1</sup>
$b = 10.7287$ (9) Å	$T = 100$ (2) K
$c = 17.3888$ (9) Å	Prism, yellow
$\beta = 130.093$ (1)°	0.35 × 0.20 × 0.08 mm
$V = 3260.9$ (4) Å <sup>3</sup>	

## Data collection

Bruker SMART CCD area-detector diffractometer	8502 independent reflections
$\omega$ scans	6899 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.048$
54767 measured reflections	$\theta_{max} = 29.6^\circ$

## Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0801P)^2 + 3.519P]$
$R[F^2 > 2\sigma(F^2)] = 0.059$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.157$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.98$ e Å <sup>-3</sup>
8502 reflections	$\Delta\rho_{min} = -0.38$ e Å <sup>-3</sup>
414 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	with 3899 Friedel pairs
	Flack parameter: 0.00 (9)

**Table 1**

Hydrogen-bond geometry (Å, °).

Cg is the centroid of the tosyl ring

D—H...A	D—H	H...A	D...A	D—H...A
C19—H193...O6 <sup>i</sup>	0.98	2.48	3.459 (3)	173
C6—H61...O6 <sup>i</sup>	0.99	2.55	3.460 (5)	153
C31—H31...O1 <sup>ii</sup>	0.95	2.56	3.379 (4)	145
C1—H11...Cg <sup>ii</sup>	0.99	3.05	3.950	153

Symmetry codes: (i)  $x, y + 1, z$ ; (ii)  $-x + \frac{1}{2}, y - \frac{1}{2}, -z + 1$ .

The absolute configuration was assigned based on the synthetic pathway, which used a compound containing a chiral reference molecule of known absolute configuration. It was further confirmed by anomalous dispersion effects in diffraction measurements on the crystal, with a Flack parameter (Flack, 1983) value of 0.00 (9) based on 3899 Friedel pairs. All H atoms were placed in their theoretical positions and treated as riding on their parent atoms, with C–H distances of 0.95 (aromatic), 0.98 (CH<sub>3</sub>), 0.99 (CH<sub>2</sub>) and 1.00 Å (CH), and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . The C11–O5 group is statistically disordered over two positions and these were refined with a fixed site occupancy of 0.5.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE-Plus* (Bruker, 1998); data reduction: *SAINTE-Plus*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The École Centrale de Paris is gratefully acknowledged for a Visiting Professorship awarded to one of the authors (NB).

The authors thank Professor Noureddine Ghermani for fruitful discussions.

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