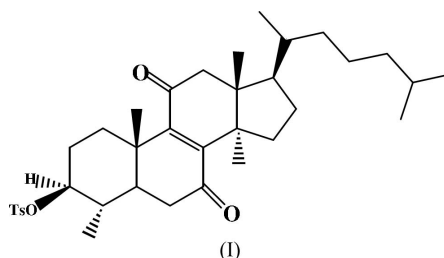


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## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å  
 $R$  factor = 0.056  
 $wR$  factor = 0.156  
Data-to-parameter ratio = 16.4For 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 $\alpha$ ,14 $\alpha$ -Dimethyl-  
3 $\beta$ -tosyl-5 $\alpha$ -cholest-8-ene-7,11-dione**The stereochemistry as well as the absolute configuration of  
the title compound,  $\text{C}_{36}\text{H}_{52}\text{O}_5\text{S}$ , were established and  
confirmed by single-crystal X-ray diffraction.Received 22 March 2005  
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## Comment

With the aim of developing and enhancing the value and  
usefulness of Moroccan natural products, we have undertaken  
the partial synthesis of terpenes with potential pharmaco-  
logical activities (Akihisa *et al.*, 1996; Smith *et al.*, 2001). The title  
compound, (I), was obtained by oxidation of 4 $\alpha$ ,14 $\alpha$ -dimethyl-  
3 $\beta$ -tosyl-5 $\alpha$ -cholest-8-ene, (1), a triterpene derivative isolated  
from the latex of *Euphorbia officinarum* (Benharref &  
Lavergne, 1985; Mazoir *et al.*, 2005). The structure of (I) was  
established by <sup>1</sup>H and <sup>13</sup>C NMR and confirmed by its single-  
crystal X-ray structure.The core of the molecule is composed of three six-  
membered and one five-membered fused rings (Fig. 1),  
forming an extended sheet parallel to the *ab* plane. The  
puckering parameters for the five-membered ring are  $q_2 =$   
0.484 (3) Å and  $\varphi_2 = 197.2$  (4)°, this latter being characteristic  
of a twist conformation (Cremer & Pople, 1975). The three  
adjacent six-membered rings C8/C9/C11–C14, C5–C10 and  
C1–C5/C10 adopt half-boat [spherical polar puckering angles  
 $\theta$  and  $\varphi$  are equal to 49.2 (3) and 239.5 (4)°, respectively], half-  
boat [ $\theta = 45.0$  (3)° and  $\varphi = 24.4$  (5)°] and chair [ $\theta = 4.3$  (3)°]  
conformations, respectively (Cremer & Pople, 1975).Atom H5 on atom C5 is involved in a C–H... $\pi$  interaction  
with the aromatic ring of the tosyl group of a symmetry-  
related molecule (symmetry code:  $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$ ; Fig. 2 and  
Table 1).

## Experimental

The tosylation and oxidation by chromic anhydride of 4 $\alpha$ ,14 $\alpha$ -  
dimethyl-3 $\beta$ -tosyl-5 $\alpha$ -cholest-8-ene, (1), isolated from the latex  
of *Euphorbia officinarum*, led to the preparation 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-one, (2), and the title compound, (I), with respective yields of  
35 and 65% (Mazoir *et al.*, 2004). Suitable crystals were obtained by

evaporation of a hexane/dichloromethane solution at 277 K.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , p.p.m.):  $\delta$  4.08 (H-3, *ddd*,  $J_1 = 11$  Hz,  $J_2 = 11$  Hz,  $J_3 = 3$  Hz), 7.77 (2H-2', *d*,  $J = 8.8$  Hz), 7.31 (2H-3', *d*,  $J = 8.8$  Hz), 2.43 (H3-5');  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , p.p.m.):  $\delta$  33.50 (C-1), 26.2 (C-2), 86.5 (C-3), 34.9 (C-4), 48.6 (C-5), 39.4 (C-6), 200.50 (C-7), 151.4 (C-8), 150.7 (C-9), 38.2 (C-10), 202.7 (C-11), 51.5 (C-12), 47.7 (C-13), 47.5 (C-14), 32.5 (C-15), 27.5 (C-16), 49.5 (C-17), 16.5 (C-18), 16.4 (C-19), 36.4 (C-20), 18.5 (C-21), 34.8 (C-22), 28.4 (C-23), 39.5 (C-24), 31.8 (C-25), 21.5 (C-26), 22.5 (C-27), 14.5 (C-30), 26.2 (C-32), 144.3 (C-1'), 134.5 (C-4'), 129.8 (C-2'), 127.5 (C-3'), 21.4 (H3-5').

#### Crystal data

$\text{C}_{36}\text{H}_{52}\text{O}_5\text{S}$	Mo $K\alpha$ radiation
$M_r = 596.84$	Cell parameters from 16 425 reflections
Orthorhombic, $P2_12_12_1$	$\theta = 1.8\text{--}26.0^\circ$
$a = 11.2528$ (2) $\text{\AA}$	$\mu = 0.14$ $\text{mm}^{-1}$
$b = 14.3549$ (3) $\text{\AA}$	$T = 293$ (2) K
$c = 20.7998$ (5) $\text{\AA}$	Prism, colourless
$V = 3359.85$ (12) $\text{\AA}^3$	$0.6 \times 0.6 \times 0.4$ mm
$Z = 4$	
$D_x = 1.180$ $\text{Mg m}^{-3}$	

#### Data collection

Nonius KappaCCD diffractometer	$R_{\text{int}} = 0.049$
$\varphi$ scans	$\theta_{\text{max}} = 26.0^\circ$
Absorption correction: none	$h = -12 \rightarrow 12$
16 425 measured reflections	$k = -17 \rightarrow 17$
6358 independent reflections	$l = -25 \rightarrow 25$
5501 reflections with $I > 2\sigma(I)$	

#### Refinement

Refinement on $F^2$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.056$	$\Delta\rho_{\text{max}} = 0.33$ $\text{e \AA}^{-3}$
$wR(F^2) = 0.156$	$\Delta\rho_{\text{min}} = -0.20$ $\text{e \AA}^{-3}$
$S = 1.08$	Extinction correction: none
6358 reflections	Absolute structure: Flack (1983),
387 parameters	2648 Friedel pairs
H-atom parameters constrained	Flack parameter: 0.01 (11)
$w = 1/[\sigma^2(F_o^2) + (0.0727P)^2 + 1.1094P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

**Table 1**

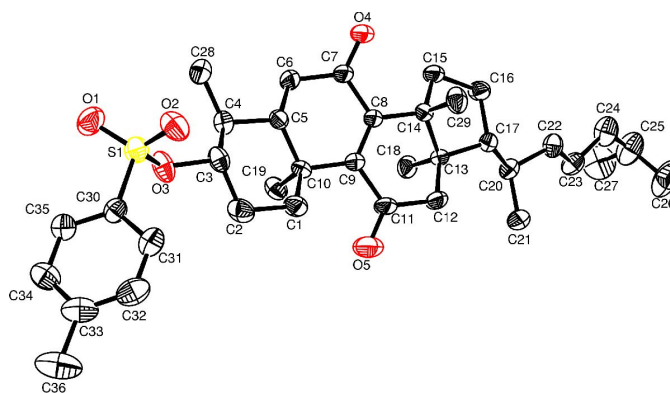
Geometrical parameters ( $\text{\AA}$ ,  $^\circ$ ) describing the C—H $\cdots\pi$  interaction between H5 and the aromatic ring of the tosyl group of a symmetry-related molecule (symmetry code:  $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$ ).

H5—ring <sup>a</sup>	C5—Cg <sup>b</sup>	C5—H5—Cg
2.96	3.981 (3)	176

Notes: (a) perpendicular distance from H5 to the ring plane; (b) Cg is the centre of gravity of the ring.

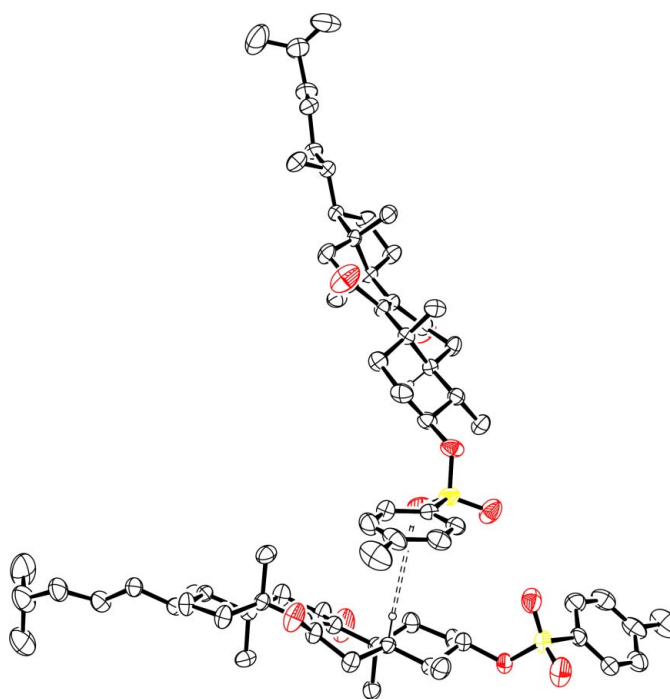
All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms. Methyl groups were allowed to rotate freely about their C—C bond, with C—H distances constrained to 0.96  $\text{\AA}$  and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ . For all other H atoms, C—H = 0.96  $\text{\AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: *KappaCCD 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).



**Figure 1**

View of the title compound,  $\text{C}_{36}\text{H}_{52}\text{O}_5\text{S}$ , showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by circles of arbitrary size.



**Figure 2**

View of the C—H $\cdots\pi$  interaction, represented as a dashed line between atom H5 and the centre of gravity of the aromatic ring of the tosyl group.

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