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Dysobinin, a tetranortriterpenoid<sup>1</sup>Sanjay Sarkhel,<sup>a</sup> Girish K. Jain,<sup>b</sup> Satywan Singh,<sup>b</sup>  
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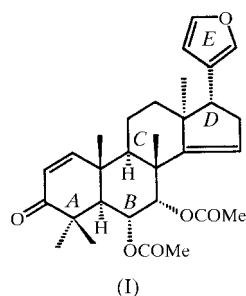
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The planar furan ring in the title compound (6 $\beta$ -acetoxyazadirone, C<sub>30</sub>H<sub>38</sub>O<sub>6</sub>) is twisted with respect to the steroid *D* ring. The crystal structure is stabilized by C—H $\cdots$ O hydrogen bonds and van der Waals interactions.

## Comment

Dysobinin is a tetranortriterpenoid belonging to the meliacin class of compounds and was isolated from *Dysoxylum binecteriferum* in around 2% yield (Singh *et al.*, 1976). It is an example of a growing family of meliacins and is of chemotaxonomic importance as it is a 6-acetoxy derivative of azadirone occurring in *Melia azadirachta*, a plant of the same family. This, together with the structural diversity of meliacin, prompted us to undertake the present study of (I).



The molecule contains one fused-ring system (*A/B/C/D*) with eight chiral centres and one furan ring (*E*). The torsion angles (Table 1) and least-squares-plane calculations (Table 2) indicate that ring *A* is a puckered sofa, while rings *B* and *C* adopt a distorted chair and a boat conformation, respectively. These three rings are *trans*-fused with each other. The cyclopentene ring (*D*) is in an envelope conformation. The furan ring (*E*) is planar,  $\alpha$ -substituted and twisted with respect to the cyclopentene ring.

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The present study does not establish the absolute configuration of the title molecule. However, based on literature precedence (Lavie *et al.*, 1971), all the triterpenoids have the methyl group at C10  $\beta$ -oriented in their absolute configuration. Accordingly, the C18 and C19 methyl groups in the title molecule have a  $\beta$  orientation, while the C20 methyl group has an  $\alpha$  orientation. The two acetoxy groups at the chiral centres, C6 and C7, whose relative stereochemistry is *S* and *R*, have  $\alpha$ -equatorial and  $\alpha$ -axial configurations, respectively. The crystal structure analysis reveals the presence of weak intermolecular C—H $\cdots$ O interactions (Table 3). These weak hydrogen-bond interactions play a significant role in the stabilization of the solid-state structure (Desiraju, 1996).

## Experimental

Dysobinin was isolated from the alcoholic extract of the air-dried powdered fruits of *Dysoxylum binecteriferum* (Singh *et al.*, 1976). Diffraction-quality crystals were obtained by slow evaporation from a methanol solution at room temperature.

## Crystal data

C<sub>30</sub>H<sub>38</sub>O<sub>6</sub>  
 $M_r = 494.60$   
 Orthorhombic,  $P2_12_12$   
 $a = 12.426$  (3) Å  
 $b = 29.446$  (10) Å  
 $c = 7.450$  (9) Å  
 $V = 2726$  (3) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.205$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 25 reflections  
 $\theta = 5.84$ – $9.98^\circ$   
 $\mu = 0.083$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Block, colourless  
 $0.30 \times 0.25 \times 0.20$  mm

## Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega$ – $2\theta$  scans  
 4725 measured reflections  
 2658 independent reflections  
 1552 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.050$

$\theta_{max} = 24.94^\circ$   
 $h = 0 \rightarrow 13$   
 $k = 0 \rightarrow 31$   
 $l = -8 \rightarrow 8$   
 3 standard reflections  
 frequency: 60 min  
 intensity decay: <0.5%

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.045$   
 $wR(F^2) = 0.127$   
 $S = 0.971$   
 2658 reflections  
 332 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0716P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.004$   
 $\Delta\rho_{max} = 0.21$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.22$  e Å<sup>-3</sup>

Table 1

Selected torsion angles ( $^\circ$ ).

C10—C1—C2—C3	1.3 (7)	C6—C5—C10—C9	56.2 (4)
C1—C2—C3—C4	–27.8 (7)	C8—C9—C10—C5	–54.0 (4)
C2—C3—C4—C5	8.4 (5)	C8—C9—C11—C12	39.7 (6)
C3—C4—C5—C10	33.5 (5)	C9—C11—C12—C13	16.1 (7)
C10—C5—C6—C7	–60.3 (4)	C11—C12—C13—C20	72.3 (6)
H5—C5—C10—C18	174.8	C11—C12—C13—C14	–52.6 (6)
C19—C8—C9—H9	175.6	C9—C8—C14—C13	17.7 (5)
O2—C6—C7—O4	61.0 (4)	C17—C13—C14—C15	–20.4 (5)
C5—C6—C7—C8	58.2 (4)	C12—C13—C14—C8	35.1 (5)
C6—C7—C8—C9	–51.0 (4)	C13—C14—C15—C16	1.9 (6)
C14—C8—C9—C11	–55.8 (4)	C14—C15—C16—C17	18.2 (6)
C7—C8—C9—C10	50.8 (4)	C15—C16—C17—C13	–29.5 (5)
C2—C1—C10—C5	39.2 (5)	C14—C13—C17—C16	30.3 (4)
C4—C5—C10—C1	–55.6 (4)	C16—C17—C30—C29	33.2 (7)

**Table 2**

Least-squares-planes data showing the deviations of atoms from the mean plane defined by atoms marked with an asterisk (\*).

Plane 1		Plane 2	
C1*	0.1239	C5*	-0.0301
C2*	-0.1614	C6*	0.0300
C3*	0.0742	C7	-0.6321
C4*	0.0322	C8*	-0.0296
C5*	-0.0689	C9*	0.0297
C10	0.6730	C10	0.6852
C18	2.2072	C18	2.2436
		C19	1.3300
Plane 3		Plane 4	
C8*	0.1045	C13*	-0.0063
C9	-0.5947	C14*	0.0119
C11*	-0.1043	C15*	-0.0121
C12*	0.1070	C16*	0.0065
C13	-0.5449	C17	0.5085
C14*	-0.1073		
C19	1.6297	Plane 6	
C20	-2.0582	C27*	0.0030
		C28*	0.0012
Plane 5		C29*	0.0005
C13*	-0.1711	C30*	-0.0021
C14*	0.0783	O6*	-0.0026
C15*	0.0499		
C16*	-0.1613		
C17*	0.2043		
C30	-0.3501		

Data collection: *CAD-4-MACH/PC* (Enraf-Nonius, 1993); cell refinement: *CAD-4-MACH/PC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *NRCVAX*; software used to prepare material for publication: *SHELXL97*.

**Table 3**

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C9—H9...O3 <sup>i</sup>	0.98	2.57	3.437 (6)	147
C20—H20C...O3 <sup>i</sup>	0.96	2.57	3.501 (6)	164
C28—H28...O5 <sup>ii</sup>	0.93	2.54	3.413 (8)	157

Symmetry codes: (i)  $x, y, z - 1$ ; (ii)  $2 - x, -y, z - 1$ .

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